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UNITED STATES
DEPARTMENT OF THE INTERIOR
HAROLD L. ICKES, SECRETARY

BUREAU OF MINES
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INFORMATION CIRCULAR

ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL, FISCAL YEAR 1937

BY

ARNO C. FIELDNER

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ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL, FISCAL YEAR 1937[1]

By Arno C. Fieldner[2]

PROPERTIES AND COMPOSITION OF AMERICAN COALS

The determination and publication of data on the chemical and physical properties of American coals have been primary functions of the Bureau of Mines since its creation in 1910.

In recent years this program has been expanded to meet increasing needs for information on the suitability of coals for the production of coke, gas, and byproducts, and within the last year an experimental coal-hydrogenation plant has been completed. With this addition, the Bureau is able to cover every phase of coal utilization, including combustion, carbonization, liquefaction, and complete gasification.

The need for this information is increasing, not only to meet the requirements of consumers but also to give the data required to establish the classification and minimum selling price of coal by the National Bituminous Coal Commission.

Fuel Inspection and Coal Analyses

During the fiscal year, 8,062 samples of coal, coke, and miscellaneous materials were analyzed in connection with the purchase of coal for the use of the Government, surveys of the national coal resources, mine safety investigations, and as a part of the research programs of the Bureau.

Two automobile trucks with equipment for mechanical crushing and quartering of samples were used in the collection of mine and tipple samples. One of these trucks completed a sampling program covering 47 mines in the State of Alabama. Both face samples and tipple samples of the various commercial sizes, as shipped, were taken. The survey included the determination of coking properties, gas and byproduct yields, friability, and grindability of the coals.

1/ The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 6992."

2/ Chief, Technologic Branch, and chief engineer, Coal Division, Bureau of Mines, Washington, D. C.

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The other truck collected samples at 11 mines in Ohio, 16 in Pennsylvania, and 73 in West Virginia.

Technical Paper 569, "Analyses of New Mexico Coals", and Technical Paper 574, "Analyses of Colorado Coals", were published,3/ and the manuscript for a Technical Paper on "Analyses of Pennsylvania Coals" is nearing completion.

The Bureau coöperated with the American Society for Testing Materials and the American Standards Association in developing standard specifications for the classification of coals by rank4/ and by grade.5/ Since July 1, 1937, all Bureau coal analyses are reported in accordance with the standard system of classification by rank. Convenient charts for calculating coal analyses to the "mineral-matter-free" basis for the purpose of classification have been developed and published6/.

Origin and Constitution of Coal

Fundamental research on the origin and constitution of coal, as revealed by the microscope in the study of thin sections and polished surfaces, has been continued in conjunction with the survey of the gas-, coke-, and byproduct-making properties of American coals. Columnar sections were examined in detail from the top to the bottom of the Clintwood7/

3/ Analyses of New Mexico Coals: Tech. Paper 569, Bureau of Mines, 1936, 112 pp.
Analyses of Colorado Coals: Tech. Paper 574, Bureau of Mines, 1937, 327 pp.

4/ Tentative Specifications for Classification of Coals by Rank (D 388-36 T): Proc. Am. Soc. Test. Mat., vol. 36, 1936, pt. 1, pp. 812-818.
Fieldner, A. C., Standardization of Coal Classification: Keystone Coal Buyers Manual, 1937, pp. 79-88.

5/ Tentative Specifications for Classification of Coals by Grade (D 389-34 T): Proc. Am. Soc. Test. Mat., vol. 34, 1934, pt. 1, pp. 841-842.
Tentative Method for Designating the Size of Coal from its Screen Analyses (D 431-36 T): Proc. Am. Soc. Test. Mat., vol. 36, 1936, pt. 1, pp. 819-822.
Fieldner, A. C., American Standard Sets Up Common Language for Coal: Ind. Standardization and Commercial Standards Monthly, vol. 7, no. 11, November 1936, pp. 273-301.
Selvig, W. A., Coal Classification, Testing, and Utilization: The Canadian Purchaser, vol. 15, no. 9, 1936, pp. 29, 31, 33, 35, 37, 39, 41, 55-56.

6/ Barkley, J. F., and Burdick, L. R., Curves for the Classification of Coal, Inf. Circ. 6933, Bureau of Mines, 1937, 6 pages.

7/ Fieldner, A. C., Davis, J. D., Selvig, W. A., Thiessen, R., and others, Carbonizing Properties and Petrographic Composition of Clintwood Bed Coal from Buchanan Mines Nos. 1 and 2, Buchanan County, Va.: Tech. Paper, 570, Bureau of Mines, 1936, 34 pp.

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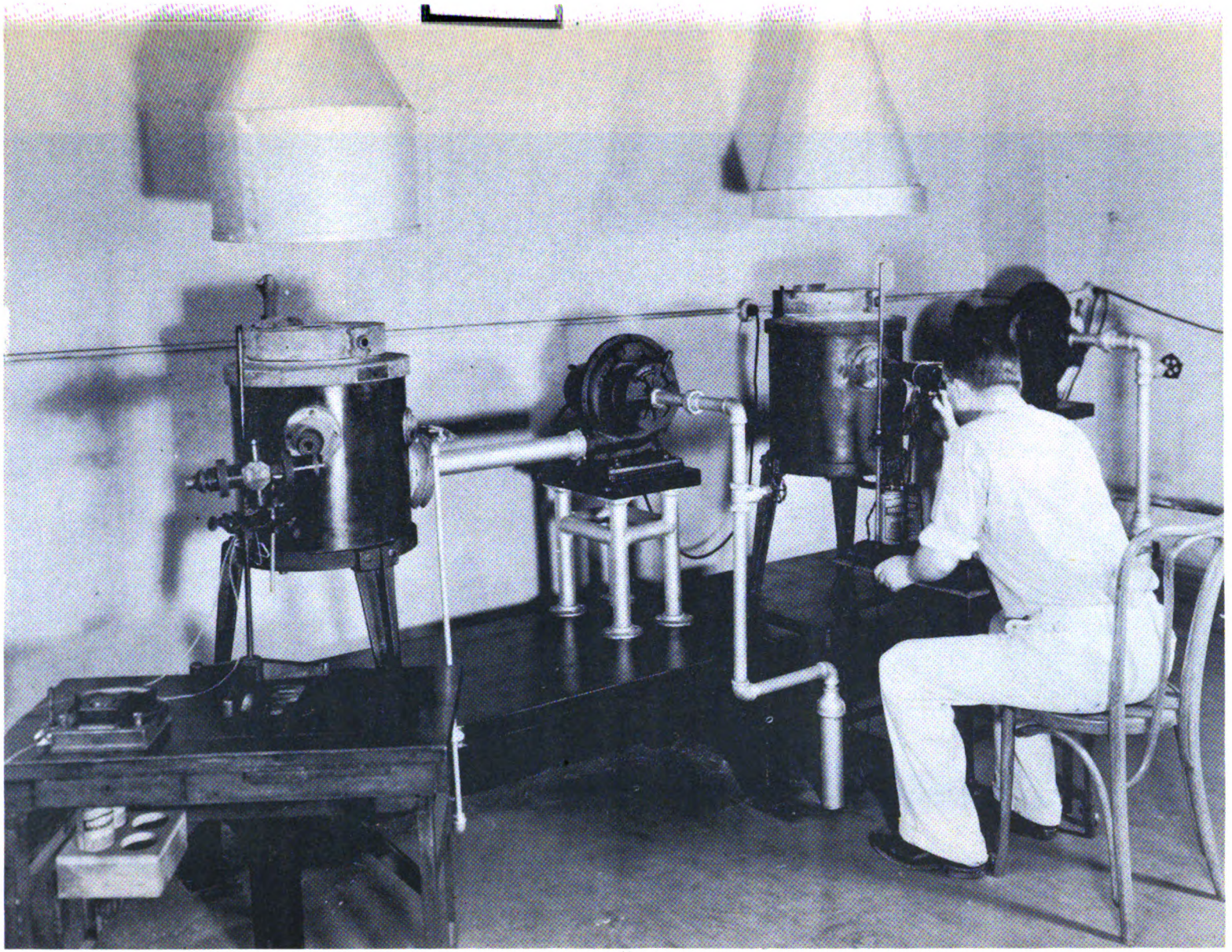

Figure 1.- Ash-fusion furnaces at Pittsburgh Experiment Station.

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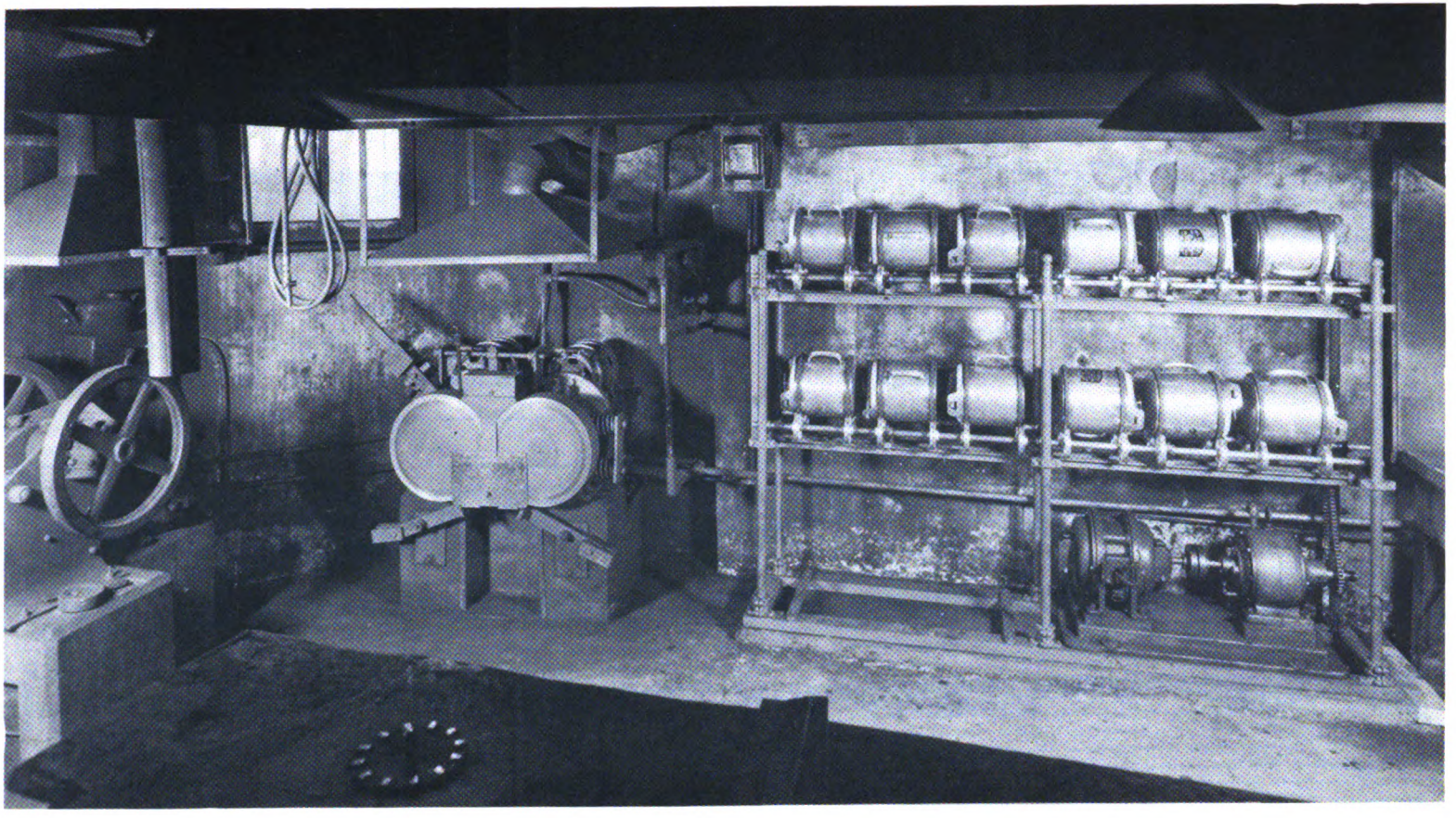

Figure 2.– Coal-sample grinding room at Pittsburgh station, showing jaw crusher on left, roll crusher in left center, and ball mill on right.

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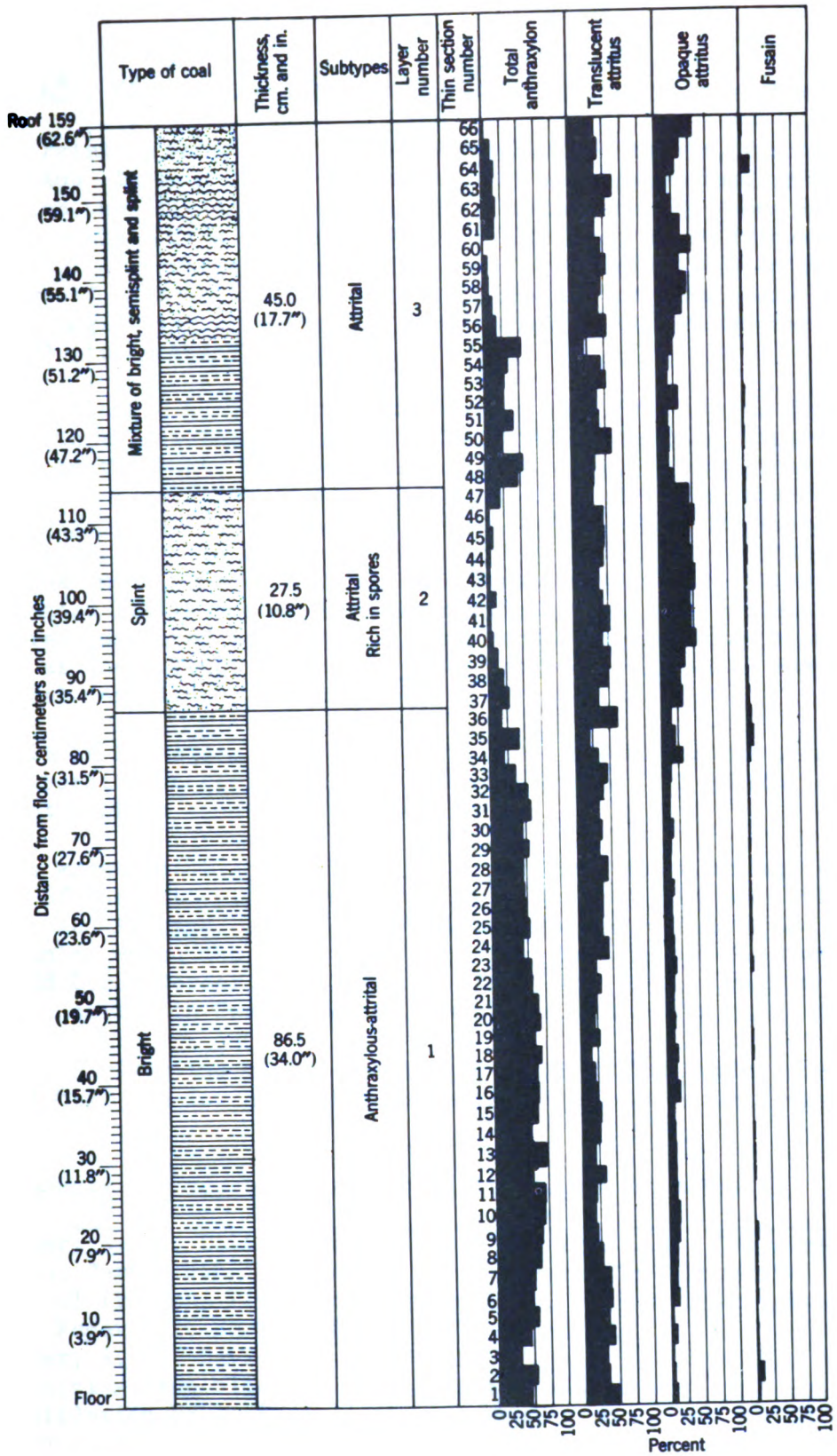

Figure 3.– Types of coal and percentage of components in Lower Cedar Grove bed, Junior mine, Mingo County, W. Va.

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Pittsburgh[8], Millers Creek[9], and Lower Sunnyside[10] beds of coal.

The relative amounts of the petrographic components, fusain, anthraxylon, and attritus, in each layer of the coal bed were determined from this examination, as illustrated in figure 3. This work has shown that most coal beds contain from two to three types of coal, which differ in chemical and physical properties. These properties were determined by separating the layers of each column and subjecting them to proximate and ultimate analyses, agglutinating tests, and assay distillation tests for gas, coke, and byproducts by the Fischer method.

Ash, volatile matter, and fixed carbon were found to be the greatest variables in the different benches. Behavior of the coal cannot be predicted on the basis of chemical analysis alone. The yields of coke and of tar were not constant, but usually the gas yield from bright coal was considerably higher than that from splint. With some exceptions (1 in 12, to date), bright coals had far higher agglutinating values than splint coals. Coke made from splint coal was dense, with few cellular spaces, and indicated little swelling, whereas bright-coal coke contained many large pores, appeared more friable, and showed two or three times as much swelling as the splint. These complete data were tabulated for possible statistical deductions.

In Table 1 the coals from the gas- and coke-making survey are arranged according to geological age. The percentage of splint, semisplint, and bright coal in each bed is given.

This arrangement shows the major importance of splint and semisplint in coals of the Pottsville series occurring in West Virginia, Virginia, Kentucky, and Alabama, and the minor importance of splint coals in Pennsylvania, which has only three important beds in the Pottsville series (Upper and Lower Mercer, and Sharon). There are 66 coal beds in the Pottsville series in West Virginia, 36 of which are of minable thickness.

Table 2 gives the chemical analyses of 12 splint coals, each from a different bed, and for comparison a series of bright coals from the same beds.

A summary of the comparative results follows in table 3.

8/ Fieldner, A. C., Davis, Davis, J. D., Selvig, W. A., Thiessen, R., and others, Carbonizing Properties and Petrographic Composition of Pittsburgh Bed Coal from the Pittsburgh Terminal No. 9 Mine, Washington County, Pa.: Tech. Paper 571, Bureau of Mines, 1936, 33 pp.

9/ Fieldner, A. C., Davis, J. D., Selvig, W. A., Thiessen, R., and others, Carbonizing Properties and Constitution of Millers Creek Bed Coal from Consolidation No. 155 Mine, Johnson County, Ky., and the Effect of Blending Millers Creek Coal With Pocahontas Bed and Pittsburgh Bed (Warden Mine) Coals: Tech. Paper 572, Bureau of Mines, 1937, 50 pp.

10/ Thiessen, R., and Sprunk, G. C., Origin and Petrographic Composition of the Lower Sunnyside Coal of Utah: Tech. Paper 573, Bureau of Mines, 1937, 34 pp.

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TABLE 1. - Types of coals in beds, arranged according to age, youngest coals at top.

Bed	State	Age	Splint	Semisplint	Bright
Sunnyside	Utah	Cretaceous	0	0	100
Pittsburgh (Edenborn mine)	Pa.	Monongahela	0	8	92
Pittsburgh (Monongah mine)	Pa.	do.	0	13	87
Pittsburgh (Avella mine)	Pa.	do.	0	0	100
Green River	Ky.	Lisman	0	0	100
Illinois No. 6	Ill. } same age	Carbondale	0	0	100
Upper Freeport	Pa. } same age	Allegheny	0	0	100
Dorothy	W. Va.	Pottsville	63	20	17
Chilton	W. Va.	do.	17	27	56
Upper Cedar Grove	W. Va.	do.	32	39	29
Lower Cedar Grove	W. Va.	do.	30	7	63
Taggart	Va. } same age	do.	6	32	62
Elkhorn	Ky. } same age	do.	16	29	55
Alma (Boone Co.)	W. Va. } same age	do.	16	0	84
Alma (Mingo Co.)	W. Va. } same age	do.	25	14	61
No. 2 Gas	W. Va. } may be same	do.	5	45	50
Millers Creek	Ky. } may be same	do.	25	14	61
Clintwood	Va.	do.	24	21	55
Upper Banner	Va.	do.	29	8	63
Sewell (Summerlee mine)	W. Va.	do.	6	13	81
Beckley	W. Va.	do.	17	6	77
Pratt	Ala.	do.	3	8	89
Mary Lee	Ala.	do.	0	0	100
Black Creek	Ala.	do.	0	0	100

Note. - The exact position of the above three Alabama coals with reference to the others in this table is not known. They belong to the middle Pottsville, which approximately corresponds to the position of the Alma coal of W. Va.

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TABLE 2. - Analyses of splint and bright coals

Coal bed	Type	Ash	Moisture- and ash-free basis					Calorific value	Softening temperature
			Proximate		Ultimate				
			Volatile matter	Fixed carbon	H	C	O	B.t.u./lb.	of ash, °F.
Chilton, W. Va.	(Bright	3.0	38.7	61.3	5.6	85.2	6.8	15,200	2740
	(Splint	9.5	35.3	64.7	5.3	95.9	6.7	15,120	2880
No. 2 gas, W. Va.	(Bright	1.5	40.2	59.8	5.7	84.5	7.4	15,110	2470
	(Splint	2.9	42.4	57.6	5.8	85.5	6.6	15,370	2490
Pratt, Ala.	(Bright	5.3	34.6	65.4	5.5	86.2	5.5	15,320	2810
	(Splint	10.8	29.4	70.6	5.1	87.5	5.4	15,320	2940+
Sewell, W. Va.	(Bright	3.0	26.6	73.4	5.0	88.5	4.3	15,520	--
	(Splint	9.3	22.6	77.4	4.6	89.0	4.6	15,350	--
Clintwood, Va.	(Bright	4.4	34.3	65.7	5.6	86.5	5.2	15,540	2910+
	(Splint	18.4	30.5	69.5	5.2	86.6	6.0	15,140	2910+
Millers Creek, Ky.	(Bright	1.0	38.9	61.1	5.4	83.5	8.8	14,770	2430
	(Splint	6.2	42.4	57.6	5.4	82.1	7.4	14,680	2170
Alma, W. Va.	(Bright	6.7	39.7	60.3	5.7	85.2	6.7	15,310	2910+
	(Splint	2.7	40.7	59.3	5.6	86.4	6.1	15,520	2910+
Upper Cedar Grove, W. Va.	(Bright	5.5	39.4	60.6	5.7	84.7	5.7	15,250	2430
	(Splint	5.4	39.8	60.2	5.6	86.1	6.0	15,330	2890
Lower Cedar Grove, W. Va.	(Bright	3.2	37.9	62.1	5.7	85.3	5.8	15,360	2520
	(Splint	4.8	35.0	65.0	5.5	87.4	5.0	15,370	2910
Beckley, W. Va.	(Bright	3.6	18.4	81.6	4.7	90.5	2.6	15,710	2910+
	(Splint	2.2	17.3	82.7	4.6	91.3	2.2	15,780	2820
Upper Banner, Va.	(Bright	3.0	32.3	67.7	5.4	87.3	5.1	15,560	2500
	(Splint	7.0	33.4	66.6	5.5	88.1	4.4	15,750	2840
Dorothy, W. Va.	(Bright	2.9	41.4	58.6	5.9	85.5	5.9	15,470	2840
	(Splint	5.6	35.2	64.8	5.3	87.0	5.5	15,390	2920+

Analyses by H. M. Cooper

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TABLE 3. - Comparison of composition of bright and splint coals

	Splint coals (number having higher value)	Bright coals (number having higher value)	Equal values
Moisture	1	9	2
Ash	9	3	0
Volatile matter	5	7	0
Hydrogen	2	9	1
Carbon	11	1	0
Nitrogen	0	12	0
Oxygen	3	9	0
Sulphur	1	11	0
Calorific value	6	5	1
Softening temperature of ash	7	2	2

In general, splint coals have the higher ash, higher total carbon, and higher softening temperature of ash; bright coals have the higher moisture, higher hydrogen, higher nitrogen, higher oxygen, and higher sulphur.

Volatile matter may vary in either direction and depends on the exact composition of the bright or splint coals, that is, whether they are spore-rich, resin-rich, etc.

Table 4 gives the yield of coke, tar, and gas and the agglutinating values of the same series of splint and bright coals.

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TABLE 4. - Agglutinating values and yields of coke and byproducts of splint and bright coals

Coal bed	Type	Percent by weight, coke	Yields per ton of coal		Agglutinating value	
			Tar	Gas	Sand: coal, ratio	weight, kilograms
			(Gals.)	(Cu.ft.)		
Chilton, W. Va.....	(Bright	74.2	32.4	2,130	16:1	5.2
	(Splint	79.1	26.2	1,670	"	3.7
No. 2 gas, W. Va.....	(Bright	72.0	33.9	2,260	"	4.1
	(Splint	70.3	44.2	1,840	"	2.9
Pratt, Ala.....	(Bright	79.1	28.1	2,050	"	6.4
	(Splint	84.3	21.6	1,560	"	1.1
Sewell, W. Va.....	(Bright	84.1	19.2	1,980	15:1	9.6
	(Splint	87.8	13.9	1,600	"	4.2
Clintwood, Va.....	(Bright	77.1	30.3	2,040	"	8.9
	(Splint	84.7	20.5	1,430	"	2.0
Millers Creek, Ky.....	(Bright	71.8	29.1	2,010	"	1.4
	(Splint	71.3	33.9	1,750	"	2.7
Alma, W. Va.....	(Bright	74.5	35.7	2,000	"	9.1
	(Splint	72.4	41.7	1,900	"	5.6
Upper Cedar Grove, W. Va.	(Bright	72.3	33.7	2,030	"	7.6
	(Splint	73.7	38.3	1,820	"	3.8
Lower Cedar Grove, W. Va.	(Bright	74.5	35.4	2,120	"	7.6
	(Splint	77.1	33.6	1,810	"	4.3
Beckley, W. Va.....	(Bright	90.7	10.0	1,730	"	9.5
	(Splint	90.4	10.7	1,830	"	4.6
Upper Banner, Va.....	(Bright	79.2	28.9	1,950	"	9.8
	(Splint	79.6	31.3	1,850	"	4.9
Dorothy, W. Va.....	(Bright	72.6	40.5	2,210	"	6.6
	(Splint	78.0	31.4	1,770	"	3.3

Analyses by W. A. Selvig and W. H. Ode.

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A summary of the comparative results follows in table 5.

TABLE 5. - Comparison of carbonization yields of bright and splint coals

Product	Splint coals (number having higher yield)	Bright coals (number having higher yield)
Coke.....	8	4
Tar.....	6	6
Gas.....	1	11

The coke and tar yield may vary in either direction, but in general the gas yield for bright coals is notably higher than from the splint coals.

The results of agglutinating tests are given in Table 4.

In all cases but one the bright coals have the higher agglutinating values. The one exception is the Millers Creek coal, which is the lowest in rank of any in the series. The bright coals from other low-rank coals also have low agglutinating values, as, for example, the bright coal from Illinois No. 6 bed, which gave no measurable agglutinating value for a 14:1 ratio of sand to coal.

Splint-coal coke, from Fischer low-temperature assay, in all cases was dense, with few cellular spaces. The coal showed little swelling.

Bright-coal coke, from the Fischer low-temperature assay, showed approximately two to three times the swelling of coke from splint coal. It contained many large pores and was more friable than the splint-coal coke.

The line of attack followed in the investigation of the origin and constitution of coal[11/] includes a study of the anatomy, structure, and chemistry of living plants, the transformation of living plants into peat through biological agencies, the composition and chemical nature of peat, the transformation of peat into coal, and the further transformation or coalification into higher ranks of coal.

Since it was discovered that the recent peats, the brown coals of the Pleistocene, the lignites, subbituminous, and bituminous coals of the Eocene and Cretaceous periods were formed essentially from conifers, the study of the anatomy and chemistry of the living conifers and their transformation into peat was essential, and therefore was undertaken as a first step.

Information was obtained on the histology, chemistry, arrangement of tissues, kinds of cell contents of the plants contributing to peat, and the reactions of microorganisms on them and their products. The various constituents composing peat were identified on the basis of this knowledge.

11/ Thiessen, R., What is Coal?: Proc. 17th Meeting Fuel Engineers, Appalachian Coals, Inc., January 1, 1937.

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The nature of a peat deposit is determined largely by the kind of plants, their cell contents (such as resins, tannins, etc.), and the degree of biological decomposition that took place. It was found also that the bark and leaves are as important, or even more important, contributors than the woody parts.

The waste products of the plant, such as the resins, tannins, and phlobaphenes, are not easily decomposed by microörganisms and have therefore concentrated to a large degree in peat. The cell walls of the tissues containing these substances have disappeared largely, whereas the resinous contents have remained almost unchanged.

Some of the tissues, particularly those of the bark and some tissues of the leaves, besides enclosing dark brown cell content, are themselves of a dark brown color, contrasting with the light color of the woody or lignified tissues. In peat these have assumed a still darker color and are distinctly of a dark brown color in contrast to the light yellow color of those constituents derived from the woody tissues.

There is but a small amount of colloidal matter in solution in peat and very small amounts of colloidal precipitates. All deposits of peat investigated have been found to be acid in nature, and hence peptonization was not possible. All colloidal solutions that may have been formed were drained off through outlets or through the soil upon which the deposit rests. Also, there are no infiltrates in the lumens of the various cells. All substances enclosed in the cell walls of peat are those of the original natural cell contents of the tissues. No further maceration of the plant matter takes place after the peat mass has been buried by subsequent debris. Chemical changes, however, do take place as the deposit matures. Microorganisms are found at all depths of the deposit; it is certain, therefore, that some of the chemical changes are brought about by microorganisms. It is possible, however, that certain chemical changes take place through purely chemical action, as by alkalies or acids.

In conjunction with the peat studies, a brown coal from the Pleistocene of Washington is being studied. This coal forms an important step between peat and lignite; both with respect to age and coalification, it is halfway between the two.

The recognition and identification of similar constituents in the brown coal are not difficult, after we have become familiar with the main constituents of peat and know their origin and the changes they have suffered through transformation into peat. It was found that the origin, composition, and general nature of this brown coal are similar to those of the peats studied.

Partly coalified remains of wood are abundant in the Pleistocene brown coal. These remains form important keys to various structures in coal, for example, the so-called "bogenstructur", the "s" structure, the dark-colored portions of remaining tracheids, and the granular opaque matter in coal.

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As a logical sequence, a lignite from the Eocene at Lehigh, N. D., was critically studied and compared with other coals of the same geological age. An interpretation of their constituents was then a simple matter. It was found that their origin and structure are similar to those of peat and the Pleistocene brown coal. Through these studies an accurate interpretation of the bituminous coals of the Cretaceous age of Utah was possible.

The outstanding observations made from the study of the younger coals are:

1. The remains of conifers form the major part of all the coals from peat to the Cretaceous bituminous coals.

2. Constituents derived from wood, bark, and leaves are represented more or less equally.

3. The cell contents form a large part of the composition of all deposits.

4. The kinds of plants that contribute determine the type of coal to a large degree, but the conditions imposed upon the deposit in early stages also determine its nature. For example, although the coals of the Eocene and the Cretaceous ages, as far as studied, are derived essentially from conifers, there are important differences in their structures and composition. Some are highly woody or anthraxylous, some are highly attrital, and others are somewhere between these extremes.

Since we have at hand a complete series of coals ranging in rank from peat to high-rank bituminous coal, formed chiefly from similar plants, a singular opportunity is presented to learn more about the processes involved in coalification. This study has only begun and the conclusions so far drawn are tentative.

There has been a progressive degree of compression with progressive coalification, but there has been no further maceration or actual effacement of structure. The fibers and cells have become more and more compressed. For this reason sections cut horizontally to the bedding plane reveal structure more completely than do cross sections. Growth rings are not at all or only barely perceptible in the anthraxylon of the bituminous coal of Utah, whereas in the fusain they are well preserved.

The knowledge obtained through the studies of transitions and progressive metamorphism of the younger coals, derived essentially from one class of plants, is directly applicable to the Paleozoic coals of all ranks. It was found that conifers were important contributors to the Paleozoic coals and that the xylem (wood), bark, and leaves have been equally important contributors. As in the younger coals, cell contents, whether of wood, bark, or leaves, have been highly concentrated. The conifers have retained their characteristics and primary structure from the older Paleozoic to the present. In addition to the conifers, the cycadophytes and the lycopods have contributed to the formation of Palezoic coals.

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No opportunity has been afforded to trace the cycadophytes from the living plant through all the stages of transition from peat to bituminous coal. Although recent cycads are available for study and their anatomy and structure are known, yet they differ so much from the ancient Paleozoic forms that they help but little. Fortunately, they have been preserved in the so-called coal balls, often in the minutest detail. Recourse was, therefore, taken to the study of the cycadophytes through coal balls. These were generously supplied by the University of Chicago through Dr. A. C. Noe. Although the study is not complete, enough has been learned of their structure to enable recognition of their remains in coals and to prove that they form an important part of many coal beds.

In the case of the lycopods, there is little in the present living forms that can be used to get adequate knowledge of the ancient forms. The living lycopods are highly specialized plants. Fortunately, again, their minute structure has been preserved in coal balls and, as in the case of the cycadophytes, recourse was had to study of the balls. In the ancient lycopods, the xylem or wood was but poorly developed. The cortex, on the other hand, was highly developed and formed the chief support of the tree, which often grew to a large size. Of the cortex, in particular, the outer part was highly specialized and consisted of thick-walled, lignified tissue resembling wood; it is called the periderm and appears to have been highly resistant to decay, and thus forms by far the larger part of the lycopod remains in coal. The stem was further covered with leaf-bases. These also appear to have been resistant to decay and were important contributors. Remains of leaves also have contributed materially. The remains of the periderms and leaf bases and leaves have retained characteristic structures and so are easily distinguishable from other plant remains.

This procedure of investigation has made it possible to determine the origin and to identify most of the constituents or components in any coal, provided sufficient structure of the tissues or other characteristics have remained. Of the conifers and the cycadophytes, the xylem, bark and leaves have remained in more less equal proportions. Of the lycopods, the periderm has furnished by far the larger part; leaves and leaf bases, however, are also prominent.

It may be shown that certain coal beds or parts of coal beds are derived predominantly from one or the other of the three great groups, conifers, cycadophytes, or lycopods. Some consist largely of the remains of one with the other two as secondary constituents. Rarely are the three represented in more or less equal proportions, and in no case has any of the three been found to be entirely absent.

Observations are being made on the primary biological decomposition of the plant matter in the peat stages. Many cases have been observed where the type or subtype of a coal is probably due to the kind and degree of decomposition. This phase is being watched throughout the whole series from peat to high-rank coal. Through a fortunate series of transitions, the origin of the granular opaque matter, one of the characterizing constituents of splint coals, was discovered[12/].

12/ Thiessen, R., and Sprunk, G. C., The Origin of the Finely Divided or Granular Opaque Matter in Splint Coals: Fuel in Science and Practice, vol. 15, no. 11, November 1936, pp. 304-15.

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In brief, coniferous wood, during the process of peat formation and further coalification, goes through a process of decay and disintegration of various intensities or degrees resulting in very small opaque particles. This granular opaque matter is the result of an advanced state of biological decomposition. Granular opaque matter is present in all coals, but is most abundant in the splint coals, where it forms one of the characterizing constituents together with the semitranslucent or brown matter.

As yet, the origin of the brown matter has not been determined satisfactorily, but all indications are that it is derived from the dark brown matters already present in the living plant. The bark of all plants contains certain tissues, the cell walls of which are of a dark brown color and are filled with dark brown cell contents. These tissues are recognized in peat as dark brown and can be traced through all ranks of coal.

Standardization of Agglutinating-Value Test for Coal

The Bureau of Mines agglutinating-value test for determination of coking and caking properties of coal was improved further by a study of various materials most suitable for use as inert material for mixing with the coal. Previous work has shown that sand is not a satisfactory inert material because of variations that occur in different shipments from the same quarry. Investigation of various manufactured materials indicates that a commercial form of silicon carbide, namely carborundum, seems to be fairly uniform in surface properties. Different lots of carborundum made at different times gave identical results with the same coal. As this material is readily available on the market, its use may solve the major difficulty in standardizing the agglutinating test method. Cooperative tests with other laboratories are in progress to determine how closely different laboratories can check in determining agglutinating values on the same coal samples. Preliminary check results with one commercial laboratory showed good agreement.

Plasticity of Coal

When a caking coal is heated rapidly over the range of temperature from approximately 400° to 500° C., it undergoes chemical decomposition, becoming more or less fused or plastic, and finally sets to form coke. The quality of the coke formed depends largely on the plastic properties of the coal and, accordingly, numerous forms of apparatus have been devised for measurement and study of these properties.

The results of about 300 tests of the plasticity of Pittsburgh and Alma bed coals made with a specially designed precision instrument show that the truly plastic properties of these coals are very transient, the life period being of the order of 1 minute at 460° C.13/

13/ Griffin, H. K., and Storch, H. H., Apparatus for Precise Plasticity Measurements at High Temperatures. Data on Coal Plasticity: Ind. and Eng. Chem., Anal. Ed., vol. 9, 1937, pp. 280-286.

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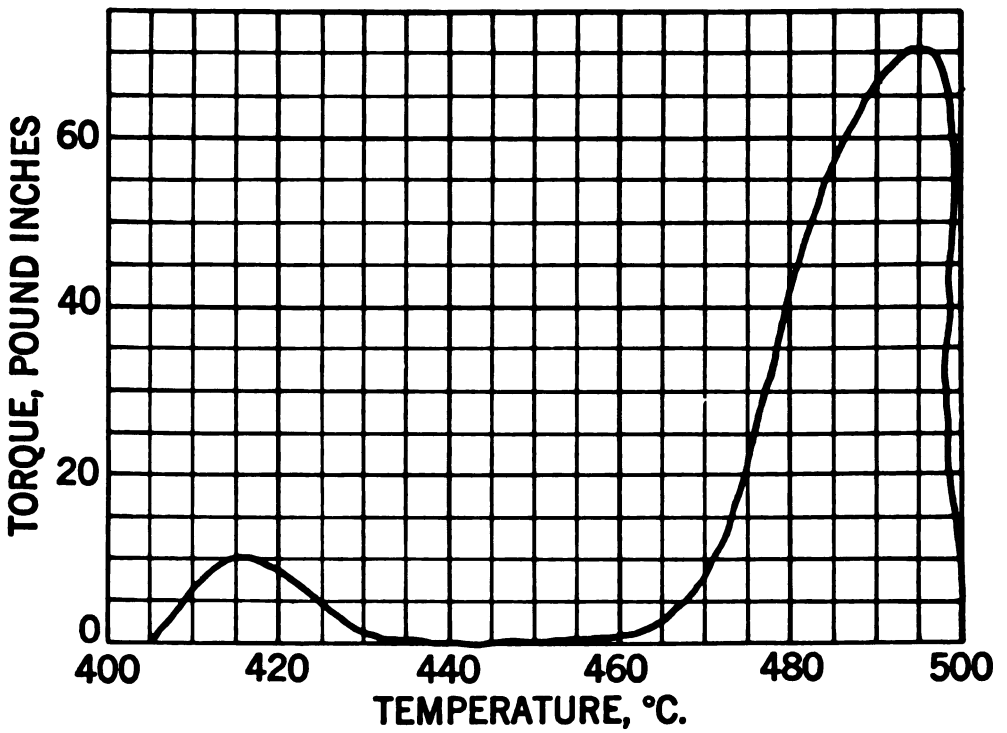

Figure 4.– Bright high–volatile A coal.

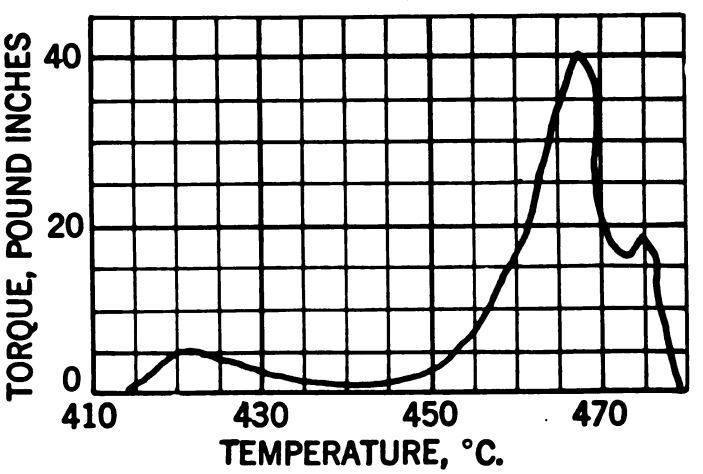

Figure 5.– High–volatile A coal containing splint.

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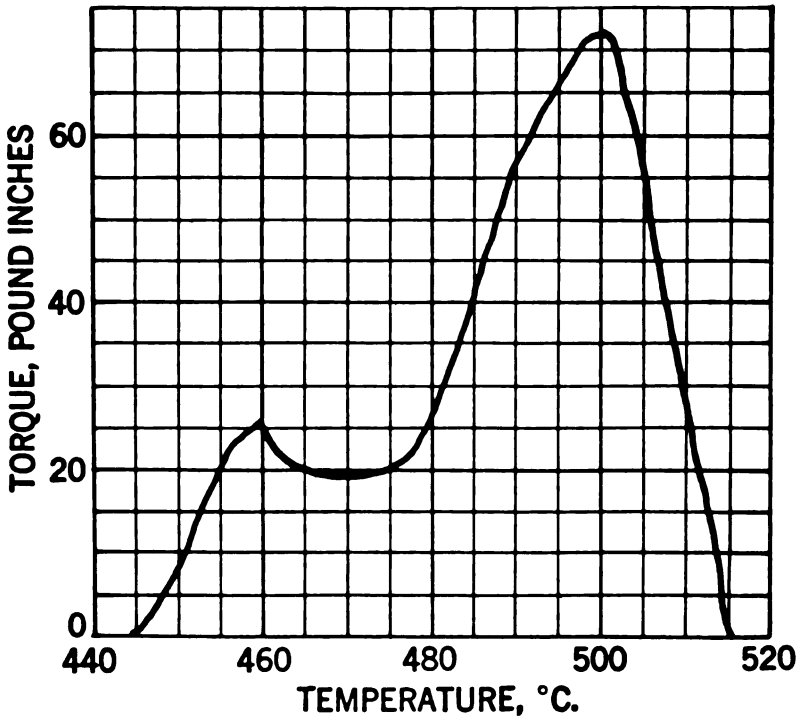

Figure 6.– Low–volatile and medium–volatile coals.

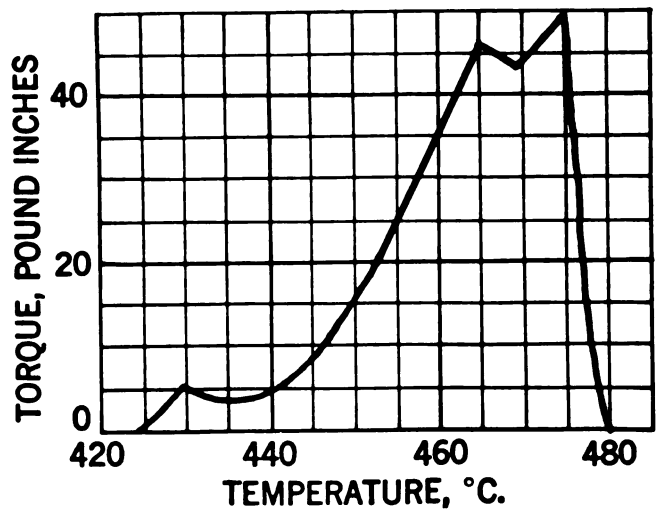

Figure 7.– Oxidized or preheated high–volatile A coal.

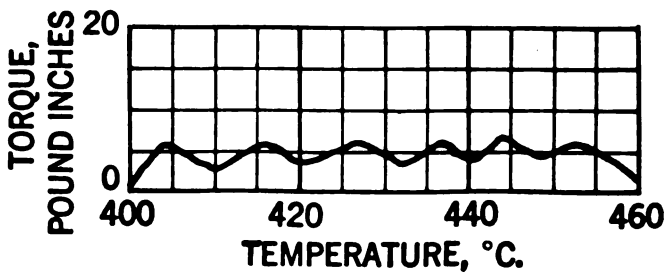

Figure 8.– Low–rank coking coals and high–rank poorly fusing coals.

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Measurements of deformations proportional to the load were obtained at 410° C. Analysis of these data indicates that the deformations are elastic rather than plastic in character. Measurements on the deformation of the solid plus oily bitumen (obtained by benzol extraction) of Pittsburgh bed coal at 120° C. show that this is a truly plastic material. However, this precision instrument does not seem suitable as yet for the practical evaluation of the plastic properties, and most of the Bureau experiments have been made with the Davis plastometer.

Results with the latter instrument have led to classification of coking coals into five groups having significantly different plastic properties. Figures 4 to 8, inclusive, show representative plastic curves for these groups. The coking property appears to depend to a considerable extent on the fusibility of the coal and particularly on the character of the region of highest fluidity as illustrated by the part of the curve from 430° to 460° C. in figure 4. This coal is too fusible (becomes too fluid) for production of coke of the best quality. It will produce a strong low-temperature coke, but when coked at high temperatures it has considerable tendency to finger and cross-fracture, and a small, weak coke with large cells results. Coals of this type are characterized as strongly shrinking. The quality of the coke can be improved by blending with a coal such as that to which the plastic curve shown in figure 6 applies, and the plasticity of the blend will be intermediate between plasticities of the constituent coals. That is, the blend coal curve will start at a higher temperature than that shown in figure 4, and the fluid range will be shorter and the minimum torque higher. Figure 5 shows the plastic curve for a high-volatile A coal containing splint. Such coals are less fusible than the type illustrated in figure 4; they also shrink less. The coke usually shows a fairly high shatter index but is likely to be abradable. The coking properties can be improved by blending these coals with low- or medium-volatile coals. Figure 6 applies to strongly coking coals of medium- and low-volatile rank. Figure 7 shows the type curve produced on mild oxidation of a high-volatile bright coal (fig. 4). The same type of curve can be produced by prolonged heating in the pre-plastic range. The effect of both methods of pretreatment is to reduce the fluidity of the coal and raise its softening temperature; and the coking properties of the coal are improved, provided treatment does not go too far. Certain blends give plasticity curves of this type. Figure 8 shows the type curve obtained from high-volatile coals of low coking rank on heating them rapidly through the plastic range. At the normal rate of heating they do not become plastic. Qualitatively, at least, this result is confirmed in coke-making practice; it is known that such coals must be coked rapidly if a good coke is to be obtained. Such a curve may also be obtained from low-volatile coals near the semi-anthracite rank. These coals do not contain enough volatile matter to produce a well-fused coke; here again, if coking is too slow, a coherent coke is not produced.

Preliminary papera on the plasticity of coal have been published[14/] and a comprehensive bulletin on this subject is being prepared.

14/ Brewer, R. E., and Atkinson, R. G., Plasticity of Coals--Its Measurement and Relation to Quality of Coke Produced: Ind. and Eng. Chem., Anal. Ed., vol. 8, no. 6, Nov. 15, 1936, pp. 443-449.
Atkinson, R. G., Brewer, R. E., and Davis, J. D., Characteristics of Coal Plasticity: Ind. and Eng. Chem., Ind. Ed., vol. 29, 1937, pp. 840-844.

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COAL MINING

The Bureau's work dealing directly with coal mining is conducted in the Technologic and the Health and Safety Branches.

The Technologic Branch conducts research and collects engineering data on mining, ventilation, explosives (including their permissibility and use), the movement of strata, the permissibility and efficiency of electrical and mechanical equipment, the mechanism of the ignition and propagation of gas and dust explosions and the development of means for their prevention, and the source of mine fires and methods for their early detection and control.

The Health and Safety Branch maintains a field force that conducts the training of miners and other workers in the mineral industries with reference to such matters as safe practices, mine rescue methods, and procedures for controlling mine fires. It educates operators and workers in the improvement and maintenance of healthful conditions, and in the extension of the use of safety devices, permissible explosives, and approved equipment. This branch cooperates in rescue work at disasters and in fighting mine fires, and it sends representatives to places where major accidents, fires, or explosions have occurred to investigate and develop information that may be used by the Bureau in its educational campaigns and research programs for the improvement of health and safety in the mining and allied industries. This branch also has charge of the gas and dust laboratories of the Central Experiment Station, Pittsburgh, Pennsylvania.

Experimental Coal Mine[15/]

Explosion tests were made of dusts prepared from samples of coal obtained from the sub-bituminous coal field north of Denver, Colo., and from the Lower Freeport bed in Jefferson County, Pa. These tests were made at the request of operators or State officials following explosion disasters.

The northern Colorado coal contains about 21 percent moisture in the mine and, because there had been no explosions in the field for many years, it had been assumed that the dust would not cause an explosion. This belief was shattered by an explosion that occurred on January 20, 1936. Large-scale tests showed that suspensions of the dust in air are about as explowive as that of the Pittsburgh coal bed, but that quite strong sources of ignition are necessary to initiate explosions. Insensitivity of the dust to weak sources of ignition is probably the reason why no explosions occurred in the field for so many years.

The coal from the Lower Freeport bed in Jefferson County, Pa., ranks close to the borderline between high-volatile and medium-volatile bituminous coals, and large-scale tests showed that its explosibility is essentially the same as would be predicted from its composition and tests of similar coals.

15/ Greenwald, H. P., Bureau of Mines Celebrates Twenty-fifth Anniversary of Its Experimental Coal Mine: Steel City News, vol. 6, no. 2, December 1936.

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Figure 9.- Visitors witnessing demonstration at Experimental mine.

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Tests were made also to determine the explosibility of dust collected from the exhaust air ducts of a vehicular tunnel. These dusts collected rather slowly during the regular course of operation of the tunnel, and physical and chemical examination showed them to be a heterogeneous mixture of ordinary air-borne dust and material that wears from automobiles passing through the tunnel. Large-scale tests showed that this dust is nonexplosive up to concentrations of 0.2 ounce per cubic foot of air space, which is approximately six times times the quantity that accumulates in the ducts per year. Regular cleaning of the ducts will eliminate any explosion hazard that may attach to this dust.

From time to time as demand arises, demonstrations of the explosibility of coal dust, of means of preventing mine explosions, and of extinguishing small mine fires, are conducted at the experimental coal mine. These tests usually are made in cooperation with the explosives testing station and are for the benefit of mining men and others who may have a particular interest in the work. Five such demonstrations were held during the fiscal year before a total of 2,200 visitors. The last of these, on June 12, was by far the largest and was attended by approximately 1,550 miners and officials from the Panhandle district of West Virginia.

The total number of visitors at the mine during the year was approximately 2,550, of whom 11 were from Canada, England, France, Germany, Italy, Japan, and Russia.

The experimental coal mine provides a number of services to the Bureau in addition to research and educational work. Most important of these during the year was the provision of means for training new personnel of the Safety Division in rescue and recovery work and in fighting mine fires. A full-scale explosion was run and the men were required to recover the mine and either rescue or recover the bodies of hypothetical men trapped by the explosion. The entire work was conducted just as it would have to be done at an explosion in a commercial mine. In addition, arrangements were made so that a large fire could be built safely in one of the passageways and the men were required to find this fire and bring it under control. The fire was sufficiently inaccessible to require sealing. This section is prepared to render similar service to the industry on request under a form of agreement that has been drafted to cover such cases.

Movement of Coal-Mine Roof

Studies of the movement of roof during progress of coal mining were carried on in one mine working in the Pittsburgh coal bed and in another working in the Thick Freeport bed, both in the vicinity of Pittsburgh. Instruments were designed to measure and record such movement accurately. Geological conditions are considerably different in the two cases. The mine working the Pittsburgh coal bed has a hard floor, and the immediate roof consists of a number of thin and weak beds of coal and shale having a total thickness of about 8 feet. Above this is a hard massive sandstone nearly 50 feet thick, the total overburden being about 300 feet thick. It was found that this sandstone is the chief factor in movements of the mine

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roof. At times, relations of roof movement to variations in the method and rate of mining could be traced, also.

The mine working the Thick Freeport bed has a fireclay floor several feet thick, and there are no hard massive strata over and close to the coal. The total overburden is about 200 feet thick. As a result of the softer nature of the strata, the behavior of the roof was different and movement was of greater magnitude. Also, there was decided upward movement of the floor in passageways close to the rib line. This movement was the result of plastic flow of the fireclay and could be measured long before it became evident to the senses. Plastic flow of the strata is more important in this case than with the rigid sandstone described above; in consequence, continued movement is more likely to occur along the rib lines during idle periods[16].

Coal-Mining Methods

Two engineers of the Mining Division of the Bureau devoted all their time to the study of mining methods and costs at individual mines or groups of mines in order to acquaint coal operators generally with improvements in practice developed locally and to increase operating efficiency and promote safety and conservation in the mining of coal.

Studies have been completed and publications issued or in preparation are on "Shaft and Slope Bottom Lay-outs at Coal Mines",[17] "Mining Methods Used in the Grundy Coal Field of Buchanan County, Virginia",[18] and "Some Aspects of Strip Mining of Bituminous Coal in Central and South Central States."[19] The latter publication is based upon studies of 18 stripping operations.

Field work has been started on concentrated, multiple-shift, mechanized coal mining in Wyoming, West Virginia, and Kentucky. The ground work has

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16/ Maize, E. R., Studies of Roof Movement in the Pittsburgh District Conducted by the Bureau of Mines: Proc. Coal Mining Institute of America: 1935-1936, pp. 123-132.
Discussion of Charles T. Holland's Paper "Pillar Deformation in a Bituminous Coal Mine", by H. P. Greenwald. This will appear in a forthcoming A.I.M.E. volume.
Greenwald, H. P., The Physics of Subsidence and Ground Movement in Coal Mines: Journal of Applied Physics, vol. 8, no. 7, July 1937, pp. 462-469.

17/ Anderson, Robert L., Shaft and Slope Bottom Lay-Outs at Coal Mines: Inf. Circ. 6949, Bureau of Mines, 1937, 39 pp.

18/ Toenges, A. L., and Anderson, Robert L., Mining Methods Used in the Grundy Coal Field of Buchanan County, Va.: Inf. Circ. 6928, Bureau of Mines, December 1936, 43 pp.

19/ Toenges, A. L., Anderson, Robert L., Some Aspects of Strip Mining of of Bituminous Coal in Central and South Central States: Inf. Circ. 6959, Bureau of Mines, 1937 (in press).

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been laid for an analysis of methods, distribution of labor, and the effect of mechanization upon efficiency, recovery of coal, safety in working, and roof control.

Fires in Anthracite Mines

Studies of the origin of fires in anthracite mines and their extinction have been continued. Analysis of the records of 170 mine fires and investigation of underground conditions lead to the following conclusions:

1. The largest number of fires are caused by explosives used in mining. Two other important causes are surface fires and revival of old fires. In general, gas explosions have not caused major fires except under abnormal gas conditions.

2. It is estimated that fires in the anthracite mines of Pennsylvania to date have cost the operators at least $50,000,000, in terms of present-day values.

3. Many fires have revived years after they were thought to have been extinguished.

4. Most fires have been extinguished by direct attack in a short time and at a slight expense.

5. The large number of revivals indicates the erratic success of indirect methods for combating fires in broken ground. Sealing has been unsuccessful generally, except as a method of retarding the spread of fires.

Laboratory studies have indicated that the susceptibility to spontaneous heating of combustible material found in mines increases with decrease of the temperature at which the rate of carbon monoxide liberation changes from a low rate to a rapidly increasing rate. This temperature, arbitrarily designated as the "carbon monoxide inflection point", is defined as the temperature at which the percent CO-time curve has a slope of 45 degrees. The ignition temperature is defined as that at which the time-temperature curve begins to depart from a straight-line heating curve.

Tests of 16 samples of coals and partings of the top split of the Mammoth vein anthracite, taken near a mine-fire area at Locust Gap, Pa., on two Washington subbituminous coals, suspected of heating spontaneously, and on a Colorado subbituminous coal, known to heat spontaneously, showed that the average"carbon monoxide inflection point"of anthracite (376° C.) was considerably higher than that of Washington subbituminous coals (220° C.) and the Colorado subbituminous coal (140° C.), known to ignite spontaneously. Extraneous combustibles, such as wood, paper, and brattice cloth, had "carbon monoxide inflection points" ranging from 170° to 260° C. Hence, these adventitious materials in anthracite mines, rather than anthracite itself, should be considered dangerous sources of spontaneous fires.

This investigation has given further proof that the presence of carbon monoxide in mine atmospheres must be regarded as indication of the existence

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of fire in the mine or at least of elevated temperature which may lead to fire.

The presence of hydrogen also indicates the existence of yet higher temperatures, since hydrogen in the laboratory tests was not evolved from oxidized anthracite until temperatures of 300° to 350° C. were reached.

Further details of this investigation are given in a Bureau of Mines Information Circular (in press), containing the annual report of the Explosives Division of the Bureau of Mines for the fiscal year 1937, and on two papers[20] published in the technical press.

Permissible Electrical Equipment

Determination of the permissibility of electrical equipment for use in mines is one of the major continuing projects of the Bureau. The duties of the testing laboratory for this work at the Central Experiment Station, Pittsburgh, have been increased from year to year owing to the rapid growth of mechanization in coal mines. The past year has been a busy one; approvals were issued on 9 coal-cutting machines, 13 loading machines and conveyors, 1 drill, 2 electric lamps, and 3 storage-battery locomotives. Two hundred seventy units now are on the approved list, and during the year more than 600 changes in design in these machines were examined and approved.

As the number of approved machines increases, the work of examining changes in design increases proportionately. Some relief from this constantly increasing demand for approvals has been obtained by examining electrical accessories and testing them for suitability for use in complete units, thereby facilitating the exchange of explosion-tested compartments among manufacturers of machinery. During the past year, eight explosion-proof compartments, including four motors, three starters, and one headlight, were investigated and "suitability" reports issued. The following publications were issued during the year:

Ilsley, L. C., Gleim, E. J., Brunot, H. B., Permissible Electrically Operated Air Compressors: Rept. of Investigations 3301, Bureau of Mines, 1936, 18 pp.

Ilsley, L. C., Hooker, A. B., and Coggeshall, E. J., The Improved Permissible Flame Safety Lamps: Rept. of Investigations 3312, Bureau of Mines, 1936, 10 pp.

Ilsley, L. C., Gleim, E. J., and Brunot, H. B., Permissible Electrically Operated Room Hoists: Rept. of Investigations 3326, Bureau of Mines, 1937, 12 pp.

20/ Scott, G. S., and Jones, G. W., Oxidation of Anthracite: Liberation of Carbon Monoxide and Its Relation to Ignition Temperature: Ind. and Eng. Chem., Ind. Ed., vol. 29, no. 1, 1937, pp. 106-8.
Scott, G. S., and Jones, G. W., Significance of Low-Temperature Oxidation Products in Anthracite Mine Fire Studies: Ind. and Eng. Chem., Ind. Ed., vol. 29, no. 7, 1937, pp. 822-826.

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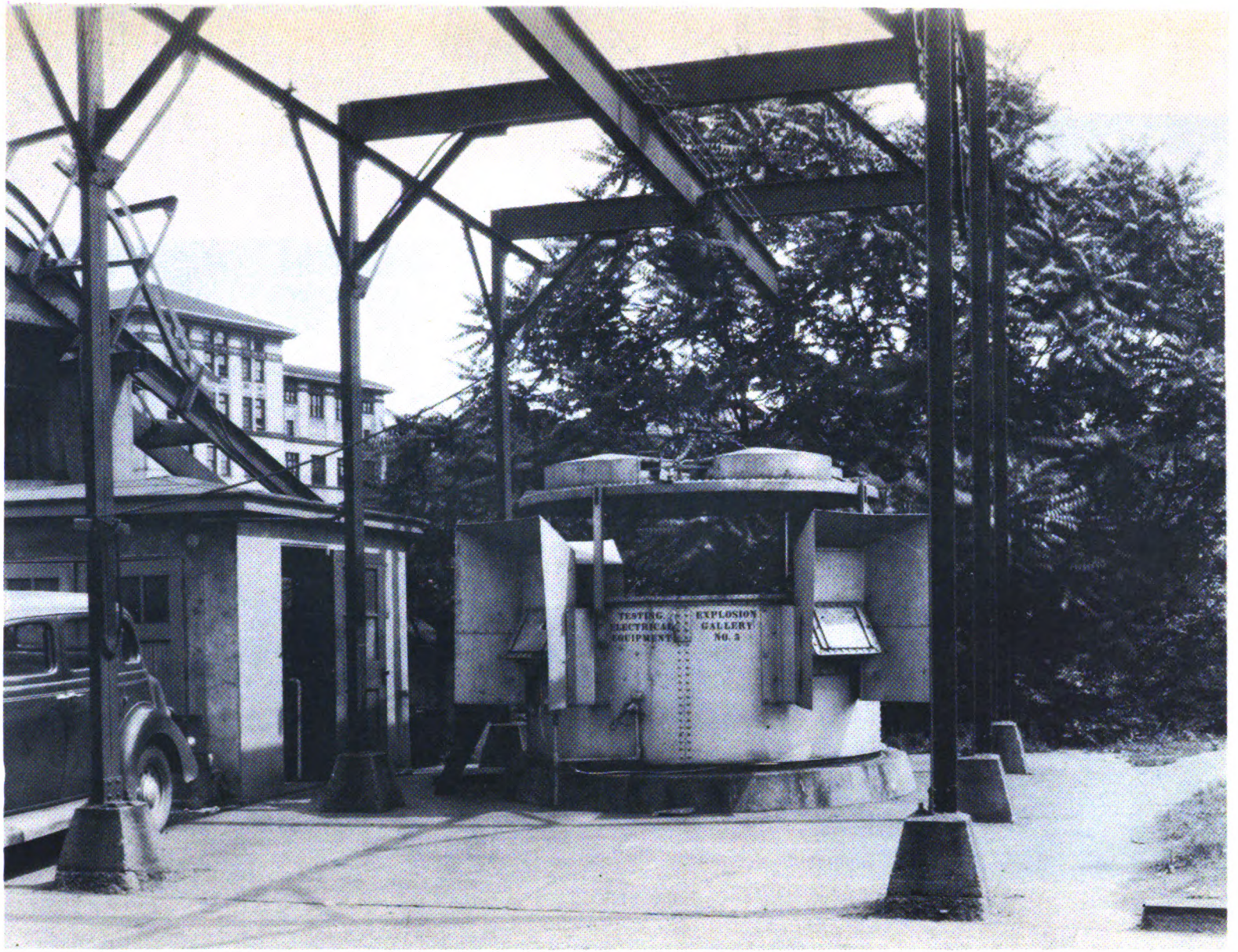

Figure 10.- Explosion gallery for testing electrical equipment.

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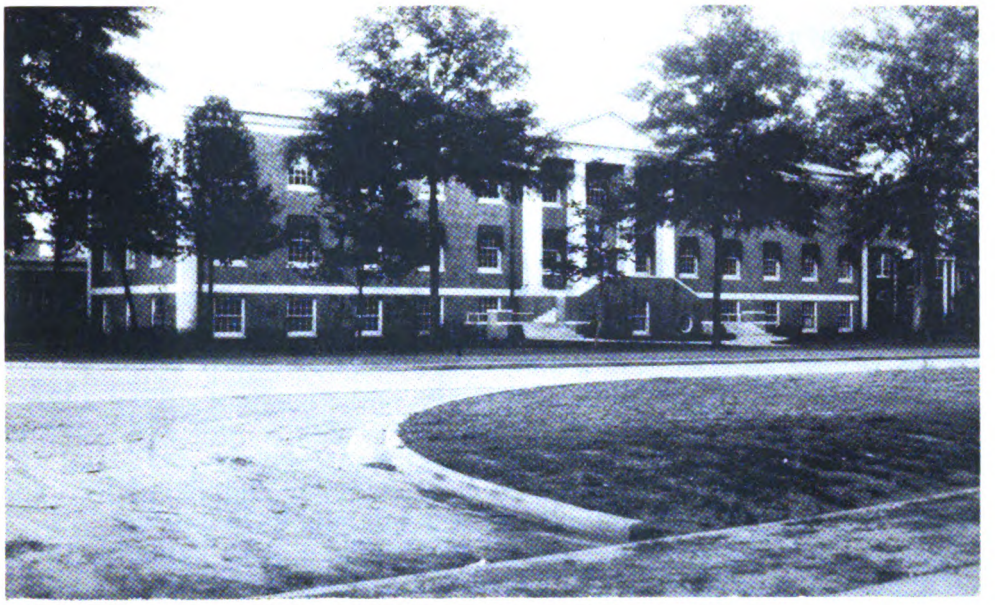

Figure 11.- Southern Experiment Station, Tuscaloosa, Ala.

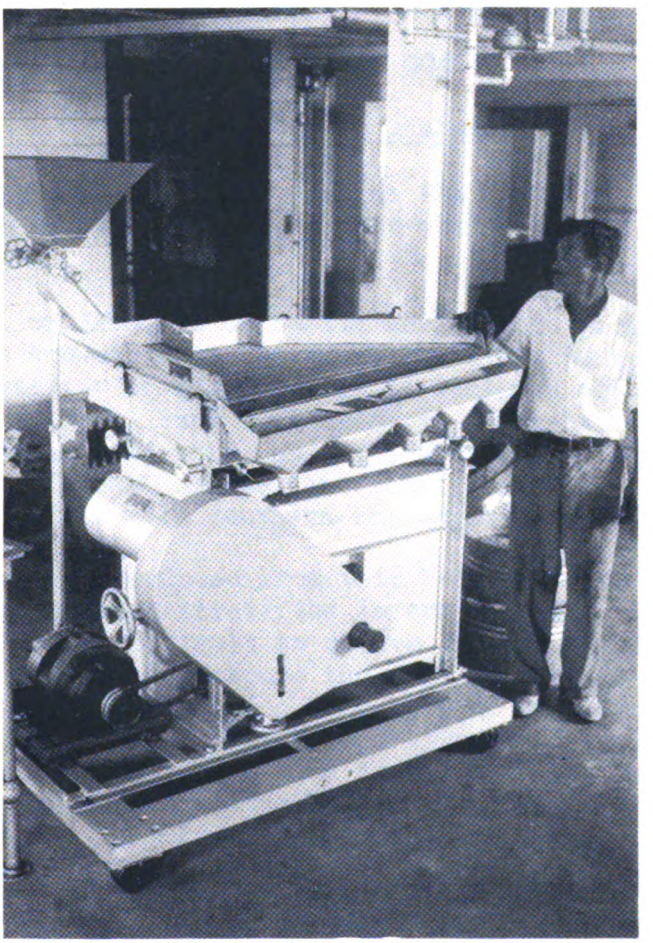

Figure 12.- Air-cleaning table. Coal-washing laboratory, Southern Experiment Station.

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Hooker, A. B., Coggeshall, E. J., and Jones, G. W., Behavior of Flame Safety Lamps in Mine Atmospheres Deficient in Oxygen: Rept. of Investigations 3327, Bureau of Mines, 1937, 6 pp.
Ilsley, L. C., Gleim, E. J., and Brunot, H. B., Permissible Electrically Operated Rock-Dust Distributors: Rept. of Investigations 3345, Bureau of Mines, 1937, 16 pp.
Gleim, E. J., Importance of Adequate Voltage for Distribution Systems in Coal Mines: Rept. of Investigations 3347, Bureau of Mines, 1937, 12 pp.
Ilsley, L. C., Milestones in Mine-safety Legislation: Inf. Circ. 6904, Bureau of Mines, 1936, 12 pp.
Gleim, E. J., Electrical Viewpoint in a Complete Safety Survey of a Coal Mine: Inf. Circ. 6894, Bureau of Mines, July 1936, 18 pp.; (Part 2) Coal Mining, vol. 13, no. 9, September 1936, pp. 9, 10.
Ilsley, L. C., List of Permissible Mine Equipment: Inf. Circ. 6942, Bureau of Mines, 1937, 23 pp.
Gleim, E. J., Bureau of Mines Apparatus for Demonstrating Electrical Ignition of Mine Gas: Inf. Circ. 6944, Bureau of Mines, 1937, 5 pp.
Schedule 12B. Single-shot Blasting Units: Requirements for Permissibility, Tests Made, and Fees Charged: 1936, 5 pp.
Schedule 16A. Multiple-shot Blasting Units: Requirements for Permissibility, Tests Made, and Fees Charged: 1936, 9 pp.

PREPARATION OF COAL

Coal Washing in Alabama

Studies on the preparation of coal at the Southern (Tuscaloosa, Ala.) Experiment Station of the Bureau were interrupted during the past year by building activities.

The allotment of $200,000 of Public Works funds permitted the construction of a new two-story and basement building, 50 by 160 feet, on the campus of the University of Alabama. It is equipped with complete facilities for coal-washing research, including crushers, screens, jigs, tables, float-and-sink testing apparatus, and laboratories for chemical analysis and physical tests of coal. These new facilities will enable the Bureau to deal more effectively with southern coal-washing problems.

In Alabama coal is produced from thin beds that contain a rather high proportion of bony coal and stratified layers of other impurities, such as slate and shale. It is difficult to remove satisfactorily the impurities from run-of-mine coal and to prepare the coal to meet the demands of a highly competitive market without a high loss of coal in the waste products.[21] During the past year, float-and-sink separations and table tests were made on the minus 1/4-inch coal from a mine in the Mary Lee bed to determine the ash reduction and recovery of clean coal suitable for byproduct coking. At another mine in the Mary Lee bed a check-up was made on the washery in

21/ Gandrud, B. W., and Coe, G. D., Washability Studies of Coal from the Henry Ellen Bed at Acmar No. 5 Mine, Acmar, Alabama: Report of Investigations 3315, Bureau of Mines, November 1936, 23 pp.

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order to determine the most practical way to reduce the ash content of the minus 3/4-inch coal so that this product could be marketed as coking coal. The desired ash reduction in this case was obtained by deepening the jig bed and passing the washed coal over a 1-1/2 millimeter slotted screen to remove some of the high-ash fines. These fines are now rewashed and then added to the steam coal.

Dewatering and Drying of Washed Coal

The dewatering and drying of washed coal has been studied. This study has consisted mainly in assembling all available literature on the subject and collecting data from plants in various coal fields where dewatering and drying are practiced most extensively. Although a considerable amount of literature has been published on this subject, both in this country and abroad, it consists mainly of scattered articles and reports appearing in a vast number of trade journals and other technical publications. In the present study an attempt is being made to prepare a fairly complete bibliography and to compile as much information as possible in the form of a publication that will be available to the industry in general.

Coal Washing in the Pacific Northwest[22/]

An investigation of the mechanism by which coal is separated from impurities by means of a full-size Baum-type jig installed in the washery at a mine was completed at the Northwest Experiment Station in cooperation with the College of Mines, University of Washington.[23/] The influence of the two principal factors - size and specific gravity - was evaluated by making screen and specific-gravity analyses of the feed, washed coal, and refuse. In this way it was possible to determine the disposition made by the jig of each size and specific-gravity component of the raw coal between washed coal and refuse.

With respect to size, the jig performed most efficiently on the intermediate range between 1-1/2 inches and 3/16 inch. The separation was less efficient on the coarsest size in the feed and least efficient on the finest particles. The overall efficiency for the entire range of sizes was 98 percent; that is, 98 percent of the material in the raw coal, which had an ash content of 10.5 percent or less, was recovered in the washed product.

The procedure used in this investigation enabled a determination to be made of the specific gravity at which washed coal was separated from refuse. At this particular washery the separation was made at 1.60 specific gravity. The degree to which the jig was able to separate the feed into

22/ Yancey, H. F., Program of Coal Research, Northwest Experiment Station, Bureau of Mines, and College of Mines, University of Washington: Coal Age, vol. 42, February 1937, p. 83.

23/ Yancey, H. F., and Geer, M. R., Performance of a Baum-Type Coal-Washing Jig: Report of Investigations 3371, Bureau of Mines, 1937, (in press).

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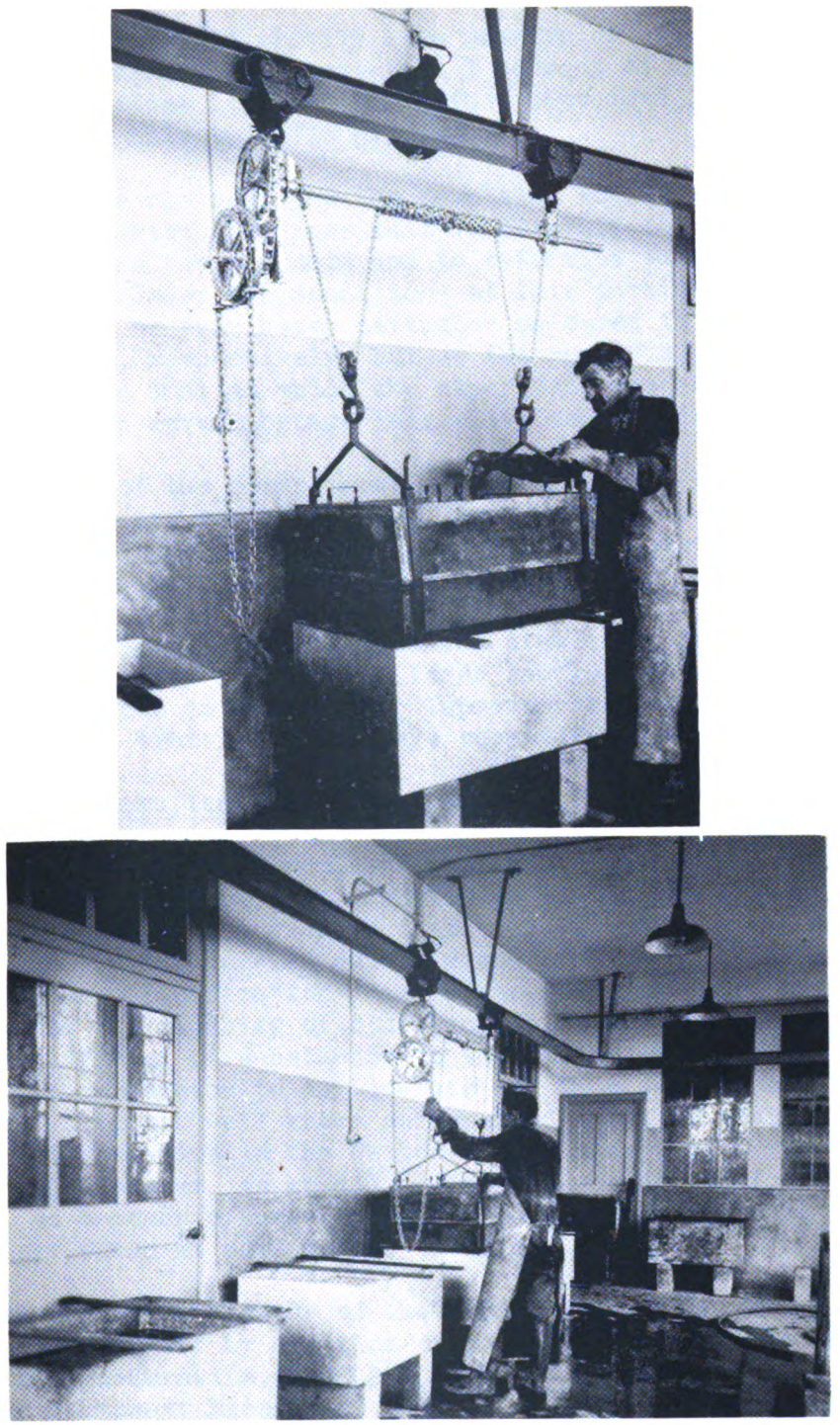

Figure 13.– Two views of float-and-sink room, showing overhead track, trolleys, and twin-hook hoist.

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two products at this specific gravity is a measure of its effectiveness. In this instance the washed coal contained 1 percent of material with a specific gravity exceeding 1.60 and the refuse contained 26.0 percent of material lighter than this specific gravity.

Separation of the raw coal and the resulting washed coal and refuse into five specific-gravity fractions made it possible to follow the course of each fraction in the two products and to determine the proportion distributed to each. Under the conditions of this particular washing operation, very light or very heavy particles appeared in the proper product, washed coal or refuse, respectively, but as the specific gravity of particles in the feed approached that at which the separation was made, the distribution to washed coal or refuse became less sharp and distinct.

A similar study of the performance of a new device for washing coal - a pulsator-type jig - has been completed recently. In the last few years several units of this type of coal-washing device have been placed in operation at mines in Indiana, Illinois, and Washington.

New equipment recently installed at the station to facilitate the making of specific-gravity analyses of large samples of coal by the float-and-sink method is shown in figure 13. Improvements consist of an overhead I-beam track on which travel two trolleys suspending a twin-hook hoist.

Clarification of Coal Washery Water

Over 40,000,000 tons of bituminous coal and probably about the same quantity of anthracite are cleaned annually in the United States by wet methods. The recovery of the fine coal and impurities from the water used for washing is ordinarily carried out by means of various forms of settling tanks. Settling and clarification may be hastened markedly, thereby greatly increasing the capacity of clarification equipment already installed, by the addition of substances to the washery water which cause the fine particles in suspension to agglomerate and thus settle more rapidly. This phenomenon, although well known and applied in other fields, has not been used at coal washeries in the United States except at a few plants. It is widely used, however, as an aid in recovery or disposal of fine coal or impurities, as a means of preventing stream pollution, and to enable re-use of washery water at many plants in Europe.

During the past year a study of the means of hastening the clarification of coal-washery water by addition of various substances to cause flocculation or agglomeration, and thus more rapid settling has been in progress at the Northwest Experiment Station. Many substances were tested for their ability to aid in this process. Both colloids and electrolytes were found to be effective under proper conditions. A higher concentration was found to be required for electrolytes than for colloids, but electrolytes, at the higher concentration, gave more nearly complete flocculation than was given by colloids at their maximum effectiveness. The most effective reagent was a colloid and an electrolyte in combination.

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None of the reagents tried could be considered suitable for all slurries. The most effective flocculent was found to be an individual problem for each particular slurry. This applied especially to the optimum quantity necessary to give good flocculation for the slurry in question. Many of the best electrolytes may have a corrosive action upon hydrolysis in washery-water circuits, so that the use of a colloidal-type flocculent alone may be the most desirable, even though it does not result in complete clarification. Of all the colloids tested, low-grade wheat flour appeared to be the most satisfactory on a cost basis in the Pacific Northwest. It was definitely the least expensive and it yielded flocculation fairly comparable to that given by any of the other substances of the same general type examined. Its action in combination with electrolytes was also very satisfactory. A mixture of equal volumes of 1 percent solutions of wheat flour and of ferric chloride was found to be an effective flocculent.

There are numerous methods of preparing "active" forms of starchy materials; one of the easiest is to heat the starch in water with or without the addition of an alkali. In addition, there are several patented processes for preparing starch. Starch from corn, potatoes, and wheat all gave good flocculents.

Tests conducted on the individual components of coal - that is, clean coal, bone coal, and clay or shale - demonstrated that the clay or shale present in many coals causes most of the difficulty in clarifying the water. Coal particles can be flocculated far more readily than the semi-colloidal particles of shale and clay.

To illustrate the settling characteristics of shale, a common constituent of many coals, as influenced by reagent concentration, figure 14 is presented. The sample used in this examination consisted of pieces of shale picked out of a lot of raw coal. These pieces were crushed dry to pass a 325-mesh sieve and were added to water to form a suspension containing 5 percent of shale. The percent of solids remaining in suspension ten minutes after varying amounts of reagent were added was determined and the results plotted against the concentration of the three reagents used. With a form of starch known as "Mogul", from 0.005 to 0.015 percent produced a maximum degree of flocculation. At higher concentrations some stabilization resulted. With ferric chloride as a reagent, a larger quantity, 0.02 percent, was necessary for the maximum degree of flocculation; but at any concentration above 0.0125 percent, it was more effective than "Mogul" with no stabilization evident. The combination of the two reagents gave better results than either of the two alone. For the same degree of flocculation, a small amount of reagent was required.

Explosion Hazards in Dust-Proofing Coal With Oil

A field investigation was made of explosion hazards at a plant where coal was treated with oil for the prevention of dust. The oil was heated to a temperature of 175° F. and sprayed at a pressure of 125 pounds per square inch on the coal as it fell from an endless conveyor upon a shaker screen. About 2 pints of oil per ton of coal was used.

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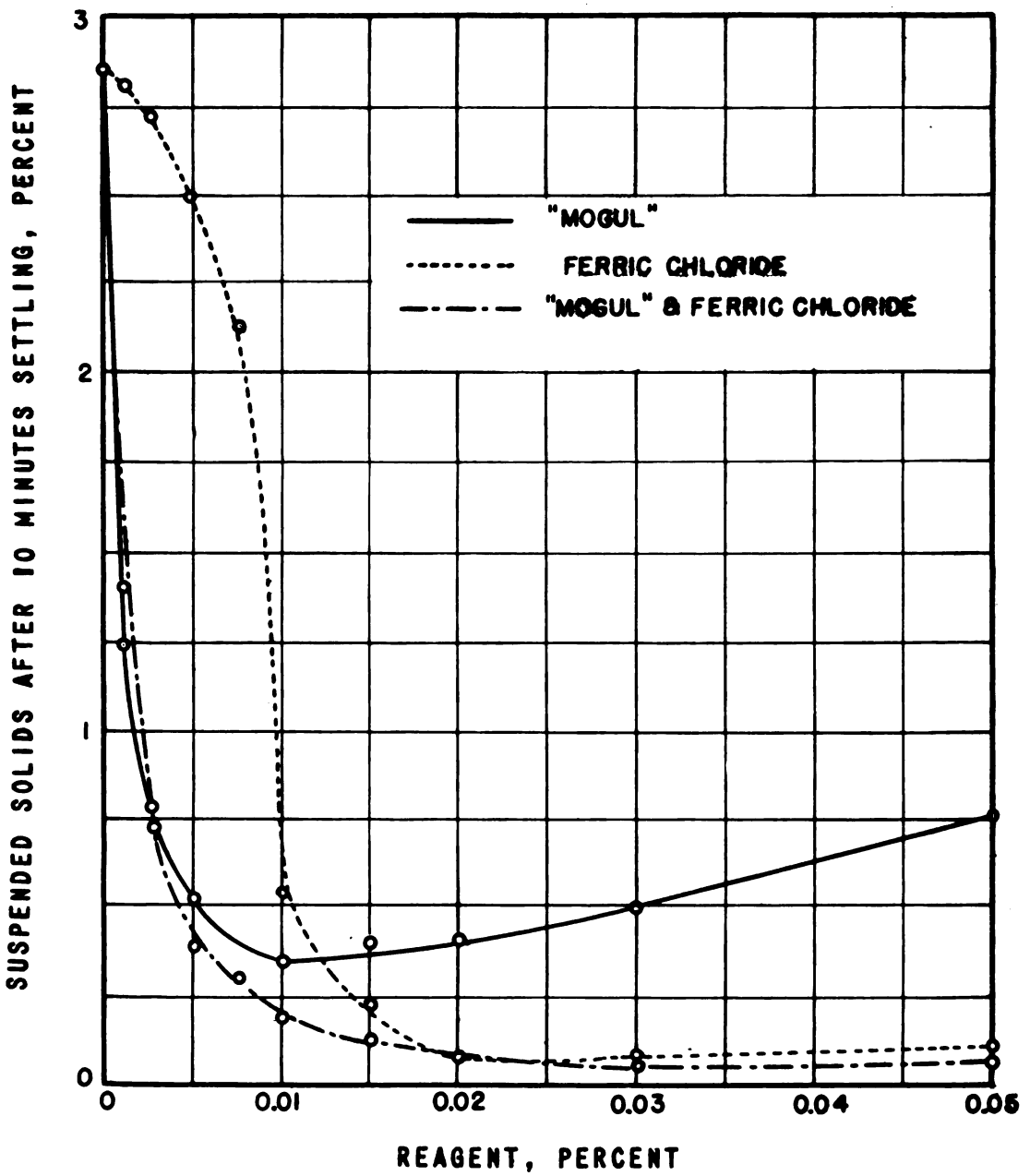

Figure 14.– Settling characteristics of shale as influenced by reagent concentration.

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The following recommendations were made to minimize fire and explosion hazards:

1. Provide a totally enclosed hood; or, if this is not possible, place ventilation ducts over an open hood to carry away any oil fog that may escape from the hood.

2. If the hoods are made of brattice cloth, they should be fireproofed.

3. The shaker screens and all machinery in the plant should be grounded to eliminate static charges.24/

4. All dust and oil deposits that collect on the floor and other parts of the plant should be removed daily, otherwise the oil concentration in the dust and the plant may become excessively high and thereby increase the fire and explosion hazard.

5. The cleaning-plant structure should be of steel and concrete. Wood or other combustible material that absorbs oil should not be used.

Effect of Dust-Proofing Coal With Oil on Rate of Oxidation

A twin calorimeter was built for studying the effect of dust-proofing treatment of coal, and preliminary experiments with a representative high-volatile C coal were made. In these experiments larger quantities of oil were sprayed on the coal than would be used in practice in order to increase the effect and render measurement positive. It is indicated that 2 to 5 percent of oil will reduce the rate of oxidation of this coal about 20 percent at 75° C., and that it makes little difference whether 2 or 5 percent of oil is used. However, it remains to be determined whether the smaller amounts of oil used in practice are likewise effective. It is also possible that different coals will be affected differently.25/

24/ Guest, P. G., Static Electricity and Petroleum Hazards: Mimeographed by Western Pennsylvania Safety Council, December 1936, 7 pp.

25/ Schmidt, L. D., Dust-Prevention Treatment of Solid Fuels: Inf. Circ. 6932, Bureau of Mines, January 1937, 10 pp.

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UTILIZATION OF COAL

The coal utilization research of the Bureau of Mines is conducted with a view to increasing fuel economy of Government power and heating plants and also for the conservation of the nation's coal resources by the promotion of more efficient utilization in general.26/

Fuel Economy Service

Upon request, this service advises Government agencies on the purchase of fuel and equipment for Government heating and power plants. Acceptance tests of new equipment and operating tests for increasing fuel economy are conducted by the engineers of this section.

Savings to the Government of approximately $25,000 were made as a result of studies made in connection with the design of these new heating plants, and operating costs at other plants were reduced materially by the introduction of improved methods of firing and the selection of more suitable fuels.

Such studies were made for the District Government, Howard University, the Capital Power Plant in Washington, and for the Veterans Administration, Department of Agriculture, Treasury Department, Federal Emergency Housing Administration, Department of Justice, and the Indian Service in various parts of the United States.

The new boiler feedwater-conditioning service has been extended to numerous Government boiler plants with resultant saving in cost of boiler operation.

26/ Fieldner, A. C., Recent Developments in Coal Preparation and Utilization: Minerals Yearbook, Bureau of Mines, 1937, pp. 935-954.
Fieldner, A. C., Advancements in Coal Research and Technology in 1936: The Mining Congress Journal, February 1937, pp. 15-16.
Rice, G. S., Fieldner, A. C., and Tryon, F. G., Conservation of Coal Resources: Reprinted by Third World Power Conference, section IV, paper no. 11, 1936, 55 pp.
Nicholls, P., Symposium on Coal Research: Discussion, Proceedings of the 19th Meeting of Fuel Engineers of Appalachian Coals, Inc., May 19, 1937.
Rice, W. E., Review of Data on Combustion in Kilns: American Ceramic Society Bull., vol. 15, no. 10, October 1936, pp. 325-330.
Rice, W. E., Factors in the Selection of Coal to be Used for the Firing of Ceramic Products (Use No. 10): Pamphlet of the Nat'l. Ass'n. of Purchasing Agents, 1937.
Fieldner, A. C., Fuels of Today and Tomorrow: Reprint of Proc. Am. Soc. Test. Mats., vol. 37, Part I, June 1937, 23 pp.

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Special attention has been given to the abatement of smoke in the City of Washington,[27] and advice on smokeless combustion[28] has been given to other municipalities that have requested aid in solving local smoke problems.

Fuel Savers Investigation

An investigation was completed of the effects on the combustion characteristics of coal and coke of pretreating them with small quantities of inorganic materials ranging in amount from 4 to 40 pounds per ton of fuel.[29]

The belief that the burning of coal and coke can be improved and that the quantity of fuel used can be reduced by the addition of chemicals has persisted for the past 100 years as shown by patent records. Householders have been sold packages of materials which, used with their coal, would act as "fuel savers." For the past 10 years the treating of coal with 1/2 to 4 pounds of mixtures of chemicals per ton of coal has been widely practiced by coal companies, but there has been lack of positive proof that any benefits have resulted to the users of the coal.

The investigations made by the Bureau were comprehensive. Experiments were made with numerous inorganic salts and other chemicals, as well as water; their effects on the burning of coals and cokes in fuel beds under different conditions were determined. The methods of burning were of necessity such that the treatment applied to the fuel was the only variable in each series of tests, so that small changes caused by the treatment could be determined and measured.

The conclusions pertaining to the use of not more than four pounds of chemicals per ton of fuel may be summarized as follows:

27/ Barkley, John F., Smoke and Fly-ash Prevention at the Federal Central Heating Plant, Washington, D. C.: Proc. 21st Ann. Convention of the Smoke Prevention Assoc., New York City, May 31 to June 5, 1937, pp. 120-128; Manual of Smoke Boiler Ordinances and Requirements, Smoke Prevention Assoc., pp. 124-127.
Nicholls, P., Duscussion on Smoke Abatement: Mimeographed Report of Proc. 16th Meeting of Fuel Engineers of Appalachian Coals, Inc., October 5, 1936, pp. 2-4.
Barkley, John F., Equipment and Operating Data on the Federal Central Heating Station: Combustion, vol. 8, no. 5, November 1936, pp. 27-32.

28/ Barkley, John F., The Forced-Draft Spreader Stoker: Trans. Am. Soc. Mech. Eng., vol. 59, 1937, pp. 259-65.
Barkley, John F., The Specific Chemical Properties of Coal as Sampled; The Hand-Fired Down-Draft Furnace; The Spreader Stoker, Natl. Assoc. of Purchasing Agents booklet entitled "Factors Recommended for Consideration in the Selection of Coal", 1937.

29/ Nicholls, P., Rice, W. E., Landry, B. A., and Reid, W. T., Burning of Coal and Coke Treated with Small Quantities of Chemicals: Bull. 404, Bureau of Mines, 1937, 158 pp.
Nicholls, P., Chemical Treatment of Coal and Coke: Tech. Pub. 848, Am. Inst. Min. and Met. Eng., 1937, 14 pp.

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The only effect on the burning that could be beneficial is the reduction of caking in the fuel beds; but any appreciable effect of this nature is limited to coals that are weakly caking, such as those from Illinois and Indiana. The action decreases rapidly as the coals are more strongly caking; it decreases rapidly with increase of rate of burning; it is approximately proportional to the amount of treatment applied; and it is small at best. Further, in most instances the same effect could be obtained by the addition of a sufficient quantity of water alone.

Treatments with chemicals, even in large quantities, do not reduce smoke nor do they in any manner reduce clinkering, but, rather, the chemicals most commonly used increase the fusibility of the ash.

No treatment can, of itself, save coal. Householders who use eastern coals can derive no benefit from treatments; those who burn weakly caking coals may, under favorable conditions, find that the furnace requires less attention.

Fusibility of Coal Ash in Slag-Tap Furnaces

Laboratory studies are being continued on the relation of ash composition and the state of oxidation of the iron to the softening temperature and the viscosity of coal-ash slags. An electric furnace with nitrogen atmosphere has been constructed for this investigation.

Combustion of Coal on Domestic Stokers

At the beginning of the fiscal year the Bureau undertook, at its Northwest Experiment Station, Seattle, Wash., where work is done in cooperation with the College of Mines of the University of Washington, a study of the combustion of coal on domestic- or residential-type stokers. The increased consumption of small sizes of coal, and particularly the rapidly increasing use of small automatic stokers, sales of which have nearly doubled each year for the past 4 years, has resulted already in a realignment of coal-preparation practice at many mines with respect to the relative quantities of the various sizes marketed.

With the object of determining the relative suitability of the various sizes and ranks of Washington coals for use on domestic stokers, the testing plant shown in figure 15 was installed. As illustrated, it consists of a conventional-style, domestic, hot-water boiler and furnace, a stoker, and the necessary auxiliary equipment, such as temperature recorders, gas-analysis recorders, draft gauges, etc., required to determine the distribution and recovery of the heat furnished on burning various sizes and ranks of coal.

The domestic stoker used almost exclusively in the Pacific Northwest, probably because of its lower first cost, is the overfeed type, and tests with different coals on this type were undertaken first. The overfeed type is considered by the manufacturers of such equipment to be suitable for burning the noncoking or weakly coking coals, such as are mined extensively in the Mississippi Valley and westward.

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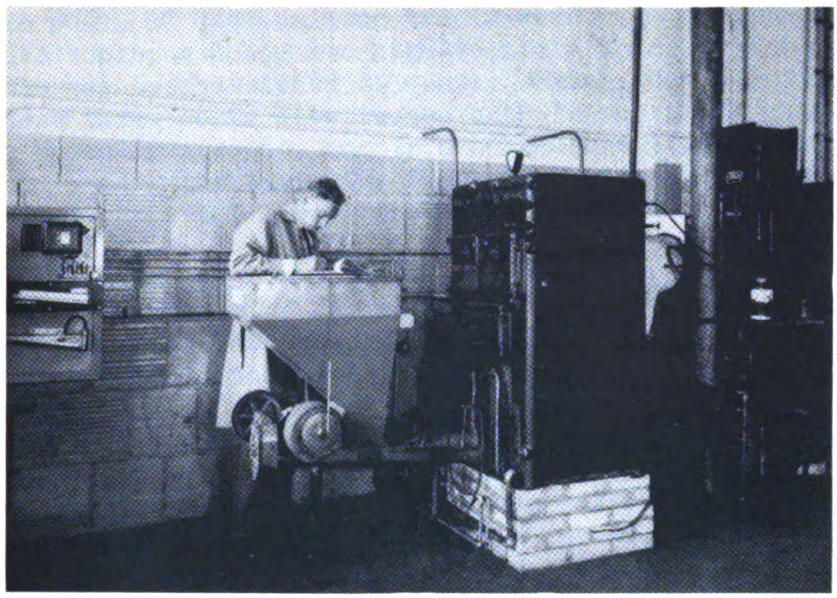

Figure 15.– Stoker and hot–water boiler furnace showing carbon dioxide analyzer and part of instrument panel.

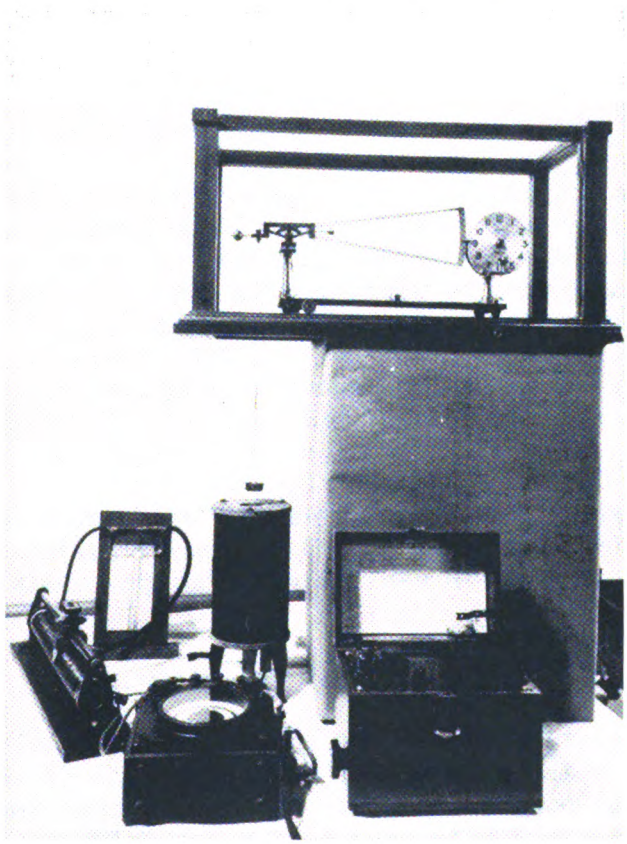

Figure 16.– Special balance and accessory apparatus for estimating combustibility of coke by direct weighing.

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During the year, 20 heat-balance trials were made on three different coals - one subbituminous A and two high-volatile bituminous A coals. Both bituminous coals may be classified as weakly caking, but one caked more than the other. These three coals were tested both in the slack sizes, including all fine sizes normally present, and in a sized condition after removal of the fine sizes. During the tests the feed rates were varied in steps from 11 to 22 pounds per hour and firing was continuous, with no "on-and-off" periods.

The general conclusions of the work done so far during the investigation, which, of course, relate only to the particular combination of stoker and boiler employed and to the conditions of testing, are as follows:

With subbituminous slack passing 1-inch, the useful heat recovered in the water ranged from 60 percent at high rates of burning to 75 percent at low rates. Thus, slack sizes of subbituminous coal, including all fine sizes and noncaking bituminous as well, are entirely suitable for overfeed-stoker use from the standpoint of over-all efficiency.

The removal from 1-inch slack subbituminous coal of the fine sizes passing a 4-mesh screen did not increase the over-all efficiency of heat recovered when burning subbituminous coal at low rates, but did increase the efficiency and reduce the ash-pit loss at high rates.

Sized, weakly caking coal with an agglutinating value of 700 grams[30/] is suitable for use on overfeed stokers; an unsized slack coal of this description has not been tested.

Coal with an agglutinating value of 2,300 grams,[30/] which also may be characterized as weakly caking, is unsuitable in the form of 1-inch slack with all the normal fine sizes present, because of the large loss of unburned coke to the ash-pit, even at low rates of burning. However, such coal can be used at low but not at high rates, if the fine sizes passing 4-mesh are removed; in other words, if the coal is sized. Under these conditions, however, the efficiency is about 15 percent less than with a noncaking coal, due principally to the high loss of coke as combustible in the ash pit.

A mixture of 50 percent subbituminous coal and 50 percent bituminous coal with an agglutinating value for the mixture of 2,300 grams,[30/] both in the form of 1-inch slack, is suitable for use at feed rates not exceeding about 15 pounds per hour.

Properties of Western Cokes

The determination of the physical and chemical properties, according to standard methods, of 20 different cokes produced locally or marketed in the Pacific Northwest has been completed, and a new method has been devised for estimating the rate at which cokes or other solid fuels burn in air.

30/ Marshall, S. M., and Bird, B. M., Agglutinating, Coking, and Byproduct Tests of Coals from Pierce County, Wash.: Bull. 336, Bureau of Mines, 1931, 31 pp.

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Figure 16 illustrates the apparatus used to estimate combustibility. A 1-gram sample of coke sized between 8- and 10-mesh sieves, amounting usually to about 250 pieces, is placed in the nichrome wire-mesh basket that is attached by a wire to the beam of the special balance shown in the photograph. The basket is then lowered into a standard electric volatile-matter furnace heated to the desired temperature, and a current of air of constant velocity is passed upward through the furnace. Continuous observation of the decrease in weight of the sample is possible throughout the entire period of burning.

For the same coke, the rate of combustion decreases with increase in the period of carbonization at a given temperature and also with increase in carbonization temperature. With cokes produced under the same conditions from coals of different rank, combustibility generally decreases with increase in rank, especially if the difference in rank is pronounced. Considering the effect of temperature on various cokes, maximum differences were shown at the lowest temperature used, namely, 600° C. At high temperatures these differences between the combustibility of various samples became less and less until, at 1,000° C., the rates were much the same for all high-temperature cokes. At this temperature, however, low-temperature cokes were still more combustible than high-temperature cokes.

Survey of the Carbonizing Properties of American Coals

In the past year several publications were issued on investigations conducted in the previous year on the gas-, coke-, and byproduct-making properties of American coals.[31] In addition, yields and qualities of carbonization products were determined on seven representative coals at

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31/ Davis, J. D., Blending Properties of Certain High-Volatile Coals as Brought Out by High-Temperature Carbonization: Proc. Am. Gas. Assoc., 1936, pp. 808-814.

Davis, J. D., and Holmes, Clarence R., Blending of Certain High-Volatile Coals with Beckley Bed Coal and the Effect on the Yield and Quality of Carbonization Products: To be published in Proc. Am. Gas. Assoc., 1937.

Fieldner, A. C., Davis, J. D., Thiessen, R., Selvig, W. A., Reynolds, D. A., Jung, F. W., and Sprunk, G. C., Carbonizing Properties and Petrographic Composition of Clintwood Bed Coal from Buchanan Mines Nos. 1 and 2, Buchanan County, Va.: Tech. Paper 570, Bureau of Mines, 1936, 34 pp.

Fieldner, A. C., Davis, J. D., Thiessen, R., Selvig, W. A., Reynolds, D. A., Sprunk, G. C., and Jung, F. W., Carbonizing Properties and Petrographic Composition of Pittsburgh Bed Coal from Pittsburgh Terminal No. 9 Mine, Washington County, Pa.: Tech. Paper 571, Bureau of Mines, 1936, 33 pp.

Fieldner, A. C., Davis, J. D., Thiessen, R., Selvig, W. A., Reynolds, D. A., Sprunk, G. C., and Holmes, C. R., Carbonizing Properties and Petrographic Composition of Millers Creek Bed Coal from Consolidation No. 155 Mine, Johnson County, Ky., and the Effect of Blending Millers Creek Coal with Pocahontas Bed and Pittsburgh Bed (Warden Mine) Coals: Tech. Paper 572, Bureau of Mines, 1936, 50 pp.

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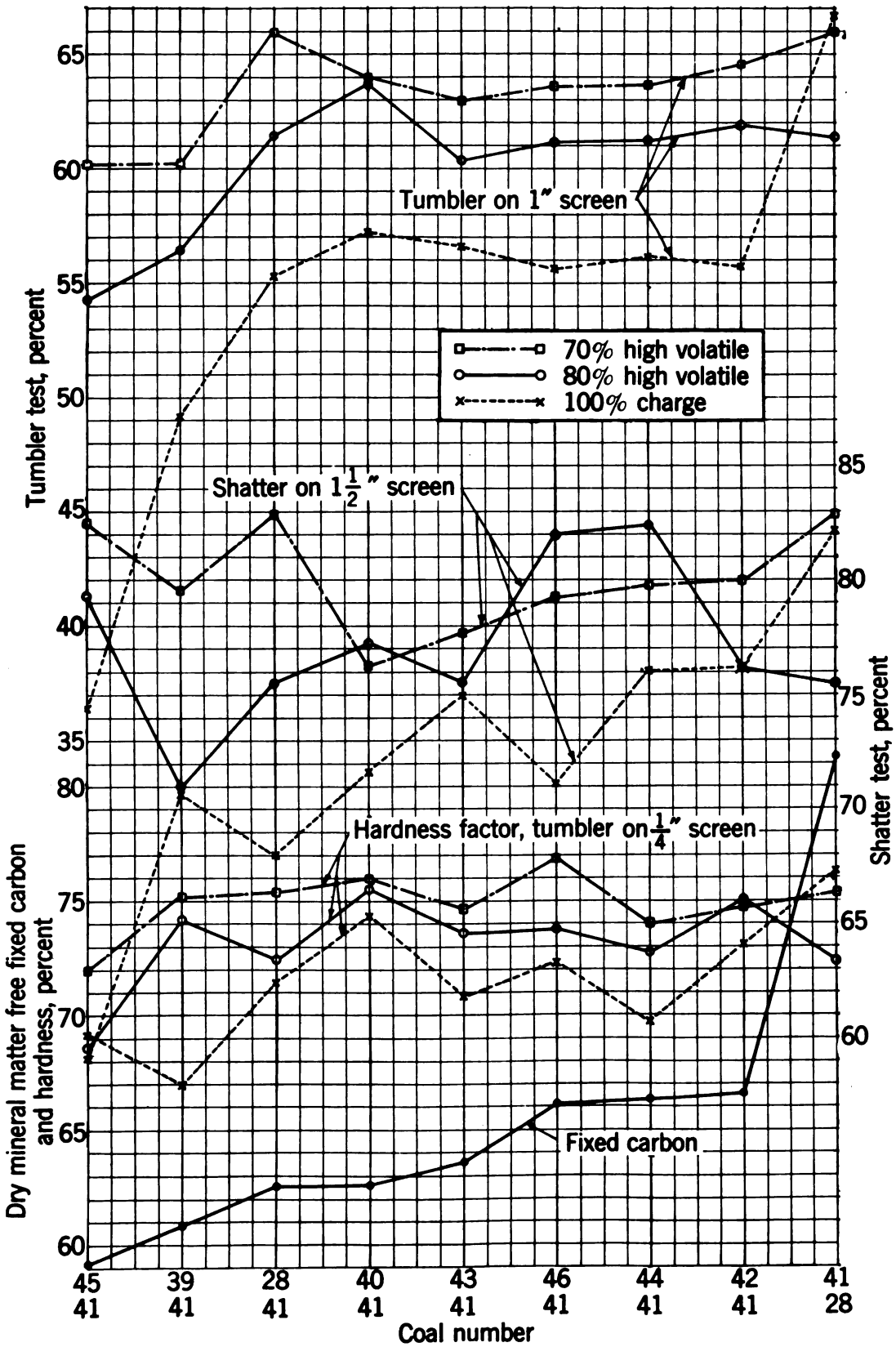

Figure 17.- Shatter and tumbler tests of cokes.

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carbonizing temperatures of 500°, 600°, 700°, 800°, 900°, and 1,000° C. Of these coals, three of low-volatile rank were from the Sewell, Beckley, and Pocahontas beds in West Virginia; two of high-volatile A rank were from the Cedar Grove and Upper Banner beds of West Virginia and Virginia; one of high-volatile C rank was from the No. 4 bed in Indiana, and the other was of subbituminous rank from Colorado.

Special attention was given to the improvement of the quality of coke by blending of different coals. All coals of high-volatile rank are blended with a single low-volatile coal, and the low-volatile coals are blended with a single high-volatile coal. The proportions used are 80 percent high-volatile with 20 percent low-volatile and 70 percent high-volatile with 30 percent of low-volatile coal. The data plotted in figure 17 show that for the coals tested (analyses and sources of the coals are given in table 6):

1. A stronger coke is obtained from a high-volatile coal blended with one of low-volatile rank than from 100 percent of the high-volatile coal, although the improvement realized is not always proportional to the amount of low-volatile coal used.

2. The improvement realized by blending a high-volatile coal of low coking rank (coal 45, table 6) is greater than for high-volatile coals of high rank.

3. The strength of coke made from a blend of 30 percent Beckley (coal 41) coal with 70 percent of Warden coal (coal 28) is practically as high as that from 100 percent of the Beckley; the hardness factor, however, is not as high.

Because most coal beds consist of alternating layers of bright coal, splint coal, and semisplint coal, a study was made of the carbonization characteristics of 15 such layers from four different coal beds. Low-temperature distillation tests and agglutinating-value tests were made. This work is informative in that it clearly shows that differences in carbonization characteristics occur for different layers of coal in the same coal bed. As a rule, the splint coal layers give lower tar and gas yields than do the bright coal layers. For some coal beds these differences are pronounced. One possible practical application of such studies is the segregation of these different layers on mining so as to divert the varieties of coal that occur in the same bed to the use for which they are best adapted. This would appear to be practicable for those coal beds composed of relatively thick layers of coal of different characteristics.

Low-temperature distillation tests at 200°, 350°, and 500° C. were made on two samples of subbituminous coal from the Denver coal region, Colorado, to determine whether these coals give off combustible matter at unusually low temperatures, as has been reported. These tests did not show any unusual characteristics for these coals[32].

32/ Ode, W. H., and Selvig, W. A., Low-Temperature Distillation Tests of Subbituminous Coal from the Denver Region Coal Field, Colorado: Rept. of Investigations 3342, Bureau of Mines, 1937, 7 pp.

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TABLE 6. - Analyses of coals, as carbonized[1/]

Coal 39 - Upper Cedar Grove bed, Junior mine, Mingo County, W. Va.
Coal 40 - Lower Cedar Grove bed, Junior mine, Mingo County, W. Va.
Coal 41 - Beckley bed, Winding Gulf No. 1 mine, Raleigh County, W. Va.
Coal 42 - Upper Banner bed, No. 9 mine, Dickenson County, Va.
Coal 43 - Dorothy bed, No. 10 mine, Kanawha County, W. Va.
Coal 44 - Powellton "A" bed, No. 9 mine, Kanawha County, W. Va.
Coal 45 - Indiana No. 4 bed, Saxton mine, Vigo County, Ind.
Coal 46 - Eagle bed, No. 3 mine, Logan County, W. Va.
Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.

Coal No.[2/]	Dry, mineral-matter-free, fixed carbon	Moist, mineral-matter-free, B.t.u.	Analyses, percent: Proximate: Moisture	Volatile matter	Fixed carbon	Ash	Ultimate: Hydrogen	Carbon	Nitrogen	Oxygen	Sulphur	Heating value, B.t.u. per pound	Softening temperature of ash, °F.
45	59.2	12,520	13.7	32.7	46.2	7.4	5.9	65.3	1.3	19.3	0.8	11,490	2,460
45A	64.4	13,590	8.5	30.6	54.1	6.8	5.4	71.6	1.5	14.0	.7	12,560	2,410
45B	66.0	13,330	10.3	28.8	54.5	6.4	5.5	70.4	1.5	15.4	.8	12,380	2,420
39	60.9	15,070	1.8	36.2	55.2	6.8	5.3	77.8	1.4	7.2	1.5	13,920	2,620
39A	65.3	15,190	1.6	32.5	59.7	6.2	5.2	79.1	1.2	7.0	1.3	14,130	2,500
39B	67.4	15,270	1.4	30.8	62.2	5.6	5.6	80.5	1.3	5.9	1.1	14,310	2,540
28	62.6	15,050	1.8	35.1	57.7	5.4	5.3	79.0	1.6	7.8	.9	14,140	2,740
41A	65.9	15,150	1.6	32.3	61.2	4.9	5.3	80.7	1.6	6.5	1.0	14,320	2,660
41B	67.8	15,220	1.5	30.5	63.1	4.9	5.2	81.3	1.6	6.1	.9	14,390	2,670
40	62.7	15,120	1.8	35.3	58.6	4.3	5.4	80.7	1.5	7.5	.6	14,400	2,800
40A	67.1	15,180	1.9	31.3	62.8	4.0	5.3	81.8	1.5	6.8	.6	14,500	2,790
40B	68.6	15,210	2.0	29.8	64.2	4.0	5.2	82.2	1.5	6.5	.6	14,530	2,780
43	63.6	15,160	2.0.	34.5	59.4	4.1	5.4	81.1	1.4	7.4	.6	14,470	2,910+
43A	66.9	15,320	1.4	31.5	62.8	4.3	5.2	82.1	1.6	6.3	.5	14,590	2,910+
43B	69.2	15,320	1.5	29.4	65.0	4.1	5.2	82.7	1.6	5.9	.5	14,620	2,910+

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TABLE 6. - Analyses of coals, as carbonized[1/] (Cont.)

Coal 39 - Upper Cedar Grove bed, Junior mine, Mingo County, W. Va.
Coal 40 - Lower Cedar Grove bed, Junior mine, Mingo County, W. Va.
Coal 41 - Beckley bed, Winding Gulf No. 1 mine, Raleigh County, W. Va.
Coal 42 - Upper Banner bed, No. 9 mine, Dickenson County, Va.
Coal 43 - Dorothy bed, No. 10 mine, Kanawha County, W. Va.
Coal 44 - Powellton "A" bed, No. 9 mine, Kanawha County, W. Va.
Coal 45 - Indiana No. 4 bed, Saxton mine, Vigo County, Ind.
Coal 46 - Eagle bed, No. 3 mine, Logan county, W. Va.
Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.

Coal No.[2/]	Dry, mineral-matter-free, fixed carbon	Moist, mineral-matter-free, B.t.u.	Analyses, percent: Proximate: Moisture	Proximate: Volatile matter	Proximate: Fixed carbon	Proximate: Ash	Ultimate: Hydrogen	Ultimate: Carbon	Ultimate: Nitrogen	Ultimate: Oxygen	Ultimate: Sulphur	Heating value, B.t.u. per pound	Softening temperature of ash, °F.
46	66.1	15,200	1.6	31.9	61.0	5.5	5.1	80.6	1.6	6.6	0.6	14,270	2,910
46A	69.2	15,160	2.3	28.9	63.6	5.2	5.1	80.8	1.5	6.8	.6	14,280	2,900
46B	70.2	15,100	2.3	28.0	64.7	5.0	5.0	81.0	1.5	6.9	.6	14,260	2,910
44	66.3	15,290	1.8	32.4	62.9	2.9	5.4	83.0	1.4	6.5	.8	14,790	2,650
44A	69.2	15,420	1.5	29.6	65.8	3.1	5.3	84.1	1.5	5.3	.7	14,880	2,700
44B	70.5	15,420	1.4	28.4	66.9	3.3	5.1	84.2	1.5	5.2	.7	14,850	2,770
42	66.6	15,450	1.6	31.8	62.3	4.3	5.3	82.8	1.4	5.4	.8	14,710	2,620
42A	69.5	15,430	1.4	29.1	65.2	4.3	5.2	83.2	1.5	5.1	.7	14,690	2,670
42B	70.8	15,420	1.4	27.9	66.6	4.1	5.1	83.5	1.5	5.1	.7	14,710	2,690
41	81.3	15,470	2.3	17.9	76.0	3.8	4.6	85.3	1.4	4.5	.4	14,820	2,919+

1/ Analyses by H. M. Cooper, chemist, Bureau of Mines.
2/ A, blends containing 20 percent Beckley; B, blends containing 30 percent Beckley.

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Effect of Conditioning Coal on its Coking Properties

Work on this problem covered three methods of conditioning, viz.: mild oxidation, heat treatment in the preplastic range, and treatment for dust prevention, the latter bearing on coking properties only incidentally, in that it may affect oxidation in storage. Most of the work of the present fiscal year has had to do with the first method. It is known that the coking power of a coal can be destroyed by severe oxidation and, conversely, that the coking power of certain high-volatile coals can be improved by mild oxidation. Presumably, with such coals one should be able to determine the optimum conditions of exposure and amounts of oxygen to use so as to obtain the best coke by this method of conditioning. With this objective in view, a suitable experimental procedure was devised. A representative sample of high-volatile A coal, 0- to 1/4-inch size, was oxidized in a thermostat at 30° C. and also at 50° C.33/ The amount of oxygen used and oxidation products formed were determined in each case and during the course of the oxidation periodic samples were withdrawn for determination of the yield and quality of carbonization products. Table 7 shows the results of the tests at 30° C. With the particular coal tested, the yield of tar decreased as the oxidation progressed, the yield of coke increased and the yield of gas decreased. As shown in table 9, the quality of the coke improved, particularly the hardness, with the amount of oxygen used.

Table 8 shows the results of a similar series of tests carried out on the same coal at 50° instead of 30° C. Under these conditions the strength of the coke (table 9) reached a maximum when the oxidation had progressed for 103 days, the oxygen content of the coal having increased from 6.7 percent to 8.3 percent (moisture- and ash-free basis). The effect of a given amount of oxygen used on the yield and quality of carbonization products appeared to be the same as that for oxidation at 30° C. Tables 7 and 8 show that the rate of oxidation at 50° C. was approximately 2-1/2 times that at 30° C.

33/ Schmidt, L. D., Elder, J. L., and Davis, J. D., Oxidation of Coal at Storage Temperatures: Ind. and Eng. Chem., Ind. Ed., vol. 28, no. 11, November 1936, pp. 1346-1353.

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TABLE 7. - Summary of 900° C. carbonization tests on oxidized Pittsburgh bed coal (Bruceton mine) at 30° C.

Sample	Av. un-oxidized	A-2	A-3	A-4	A-5	A-6	A-7
Extent of oxidation, days.....	0.0	4.4	12.4	35.3	51.8	80.0	113
Grams O_2 per kilogram dry coal	.0	.85	2.04	4.61	5.65	6.86	8.29
Coal charged to retort							
Average size[1] (cm)	.353	--	.295	.274	.292	.337	.310
Yields, percent[2]:							
Coke.....	65.1	65.0	65.6	65.9	66.2	66.0	67.0
Tar.....	13.5	13.6	12.9	12.7	12.6	11.9	11.5
Liquor.....	4.6	4.3	4.4	4.5	4.7	4.2	4.1
Gas.....	16.1	15.2	16.0	15.7	15.9	15.9	15.8
H_2S.....	.4	.4	.4	.4	.4	.4	.4
Total.....	99.7	98.5	99.3	99.2	99.8	98.4	98.8
Gas[3], liters per kg coal..	303	284	302	295	303	302	303
Gas[3], cu. ft./ton[4] coal..	9,710	9,120	9,680	9,470	9,710	9,690	9,730
Total CO_2 evolved in carbonizing (gm/kg dry coal).....	9.26	10.75	9.70	10.95	11.15	12.31	11.70
Gas analysis, percent:							
CO_2.....	1.6	2.0	1.7	1.9	1.9	2.1	2.0
Ill.....	6.1	5.2	5.9	5.6	5.8	6.4	6.2
O_2.....	.3	.3	.6	.3	.3	.4	.5
H_2.....	43.7	51.3	46.5	49.5	49.5	46.7	48.4
CO.....	6.5	6.3	6.9	6.5	6.8	6.8	6.8
CH_4.....	31.4	29.3	31.3	30.7	30.3	31.9	31.2
C_2H_6.....	2.9	3.6	3.0	3.4	3.2	2.8	3.5
N_2.....	2.5	2.0	4.1	2.1	2.2	2.9	1.4
Gas, sp. gravity[5].....	.445	.441	.439	.440	.437	.437	.435

1/ Yancey, H. F., and Zane, R. E., Comparison of Methods for Determining the Friability of Coal: Rept. of Investigations 3215, Bureau of Mines, 1933, pp. 1-23.
2/ Dry basis.
3/ Saturated at 60° F. and 30 inches mercury (15.56° C. and 762 mm Hg).
4/ 2,000-pound ton.
5/ Specific gravity air = 1.

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TABLE 8. - Summary of 900° C. carbonization tests on Pittsburgh bed coal (Bruceton mine) oxidized at 50° C.

Sample	Unoxidized B-1	B-2	B-3	B-4	B-5	B-6	B-7
Extent of oxidation, days	0.0	15.0	32.8	64.5	67.4	103.4	153.3
Grams O_2 per kilogram dry coal	.0	4.4	10.3	18.8	19.5	27.2	31.3
Coal charged to retort							
Average size[1] (cm)	.354	.284	.295	---	.323	.338	.396
Yields, percent[2]:							
Coke	66.7	67.3	67.6	---	68.3	68.9	68.1
Tar	12.3	12.4	11.1	---	9.4	8.3	8.7
Liquor	4.0	4.3	4.6	---	5.4	5.7	5.7
Gas	15.9	15.7	16.3	---	16.4	17.6	16.8
H_2S	.4	.4	.4	---	.4	.3	.3
CO_2[6]	.05	.02	.04	---	.07	.09	.08
Total	99.3	100.1	100.0	---	99.9	100.8	99.6
Gas[3], liters per kg coal	302	305	310	---	310	312	308
Gas[3], cu. ft./ton[4] coal	9,690	9,770	9,940	---	9,950	10,000	9,860
Total CO_2 evolved in carbonizing (gm/kg dry coal)	11.59	12.62	13.45	---	15.64	17.18	19.14
Gas analysis, percent:							
CO_2	1.9	2.2	2.1	---	2.5	2.6	3.1
Ill	5.5	5.4	5.4	---	5.0	5.8	5.1
O_2	.9	.5	.7	---	.7	.7	.7
H_2	49.3	50.6	50.3	---	48.6	45.5	48.4
CO	7.0	7.0	7.3	---	7.9	8.4	8.6
CH_4	30.1	29.1	28.8	---	30.3	32.5	29.2
C_2H_6	3.0	3.0	2.8	---	1.7	2.2	2.0
N_2	2.3	2.2	2.6	---	3.3	2.3	2.9
Gas, sp. gravity[5]	.430	.424	.427	---	.434	.463	.448

1/ Yancey, H. F., and Zane, R. E., Comparison of Methods for Determining the Friability of Coal: Rept. of Investigations 3215, Bureau of Mines, 1933, pp. 1-23.
2/ Dry basis.
3/ Saturated at 60° F. and 30 inches mercury (15.56° C. and 762 mm Hg).
4/ 2,000-pound ton.
5/ Specific gravity air = 1.
6/ CO_2 removed on drying at 118° C. prior to carbonization.

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TABLE 9. - Physical properties of coke produced on carbonizing oxidized Pittsburgh bed coal (Bruceton mine)

Oxidation at 30° C.

mple	Av. un-oxidized	A-2	A-3	A-4	A-5	A-6	A-7
tent of oxidation, days.....	0.0	4.4	12.4	35.3	51.8	80.0	113
ams O_2 per kilogram dry coal...	.0	.85	2.04	4.61	5.65	6.86	8.29
ke, screen analysis after hour in tumbler (cumulative):							
Percent on 1-1/4 inch.....	11.3	17.1	21.6	16.9	21.1	12.0	13.2
Percent on 1 inch.....	34.9	42.8	40.6	44.0	42.1	45.9	39.6
Percent on 3/4 inch.....	55.6	56.0	58.8	62.2	59.2	63.0	64.3
Percent on 2 Mesh.....	63.0	63.0	64.7	66.9	67.8	68.9	71.4
Percent on 4 Mesh.....	64.2	64.2	66.3	67.7	68.8	69.3	72.4
Average size[1] (cm).....	1.80	1.89	1.94	2.02	1.99	1.99	2.00
Percent Friability[1].....	57.9	55.7	54.4	52.6	53.4	53.2	53.0
ke, screen analysis after 1-1/2 urs in tumbler (cumulative):							
Percent on 1-1/4 inch.....	7.2	7.0	12.2	7.3	11.1	5.4	3.0
Percent on 1 inch.....	27.0	34.5	34.0	33.0	35.5	35.4	27.4
Percent on 3/4 inch.....	48.7	49.6	52.5	53.0	52.6	51.8	56.8
Percent on 2 Mesh.....	56.7	57.0	58.5	61.2	62.1	62.3	65.2
Percent on 4 Mesh.....	57.9	57.9	60.2	62.0	63.0	62.7	66.3
Average size[1] (cm).....	1.59	1.65	1.72	1.71	1.76	1.72	1.73
Percent friability[1].....	62.6	61.3	59.6	59.7	58.6	59.6	59.4

Oxidation at 50° C.

mple	Unoxidized B-1	B-2	B-3	B-4	B-5	B-6	B-7
tent of oxidation, days.....	0.0	15.0	32.8	64.5	67.4	103.4	153.3
ams O_2 per kilogram dry coal...	.0	4.4	10.3	18.8	19.5	27.2	31.3
ke, screen analysis after hour in tumbler (cumulative):							
Percent on 1-1/4 inch.....	13.3	15.6	17.2	--	6.5	12.7	7.4
Percent on 1 inch.....	40.8	34.4	42.2	--	40.4	37.9	46.5
Percent on 3/4 inch.....	57.8	61.4	64.0	--	68.0	68.4	64.5
Percent on 2 Mesh.....	65.9	69.6	69.4	--	75.8	73.7	72.6
Percent on 4 Mesh.....	67.2	71.4	70.9	--	76.7	74.5	73.6
Average size[1] (cm).....	1.897	1.946	2.047	--	2.047	2.047	2.024
Percent friability[1].....	55.5	54.3	51.9	--	51.9	51.9	52.5
oke, screen analysis after 1-1/2 ours in tumbler (cumulative):							
Percent on 1-1/4 inch.....	3.2	7.7	12.0	--	2.3	3.7	2.0
Percent on 1 inch.....	30.7	26.7	33.0	--	33.0	27.1	37.3
Percent on 3/4 inch.....	50.0	53.0	56.4	--	60.5	62.3	57.3
Percent on 2 Mesh.....	59.9	63.0	62.6	--	70.6	68.3	67.0
Percent on 4 Mesh.....	61.2	65.0	64.1	--	71.5	69.3	67.9
Average size[1] (cm).....	1.638	1.704	1.791	--	1.854	1.814	1.811
Percent friability[1].....	61.5	60.0	58.0	--	56.5	57.4	57.5
Apparent specific gravity of coke	.774	.774	.739	--	.807	.838	.808
True specific gravity of coke.	1.902	--	1.854	--	--	1.867	--
Percent cell space.....	59.3	--	60.1	--	--	55.1	--

[1] Yancey, H. F., and Zane, R. E. (see footnote, table 6).

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The Swelling of Coals During the Coking Process

When bituminous coal fuses and becomes plastic in the coking process, it expands because of the formation of gases within the plastic mass, much the same as dough rises in the process of bread making. Various laboratory methods have been devised to study the expansion of coals in the plastic state and to classify them as to expanding properties, but none of them is of much practical value for determining how a coal will expand in industrial carbonizing apparatus. In these test methods a small quantity of fine coal is used, so that the whole can be heated uniformly, whereas in industrial practice only a thin layer perpendicular to the direction of flow of heat is plastic at one time and this layer gradually advances to the center of the heating chamber. The plastic layer varies in thickness as the carbonization progresses over the 7 to 12 inches of the charge and, therefore, the freedom of escape of the gases generated therein varies. Accordingly, the expansive force varies so that conditions of test should duplicate those in industrial ovens if the results are to be of practical application. One test method involving the progressive heating idea has been used in the United States for several years, but because of the small scale of test the results have been questioned. For this reason, a larger-scale apparatus more nearly duplicating a coke oven was designed and built. Views of this apparatus in open and closed positions are shown in figures 18 and 19. It is a slot-type oven, heated electrically on one wall only, the opposite wall being movable so that the amount of expansion of the coal being coked can be measured while a constant pressure is being maintained on the wall. The normal thickness of the charge (200 pounds) is 5 inches, but it can be increased to 8 inches if desired. In addition to this piece of apparatus, a smaller oven (holding about 40 pounds) was built following a design recently adopted by a steel company with which the Bureau is cooperating. This oven is heated electrically from the sole; it is shown in figure 20.

Previous investigators have shown that height and density of the charge affect the amount of expansion of a coal being coked under industrial conditions. These factors are known for coke-oven practice and can be controlled in test apparatus. The first experimental work with the Bureau of Mines apparatus showed that rate of heating, particularly that through the plastic range of temperature, has a pronounced effect on the expansion obtained. For example, it was shown, for a strongly expanding coal, that by doubling the average rate of heating through this range the expansion under constant load was doubled. It was evident, therefore, that coke-oven heating rates would have to be maintained in the test apparatus if results directly applicable to industrial coking were to be obtained. A study of published data on the heating of charges in coke ovens was made, and it was found that average rates of heating vary from about 2 to 3.5° C. per minute, depending on the flue temperature and width of the oven. Since the Bureau of Mines test ovens are heated electrically, accurate control is possible. Work done so far indicates that the average rate of heating through the plastic range in a coke oven can be duplicated without having the rates excessively high or low at any point. It is indicated that on duplicating coke-oven carbonizing conditions as regards heating, charge density, and pressure on the charge, the test results give a true picture of the expansive behavior of coal in a coke oven.

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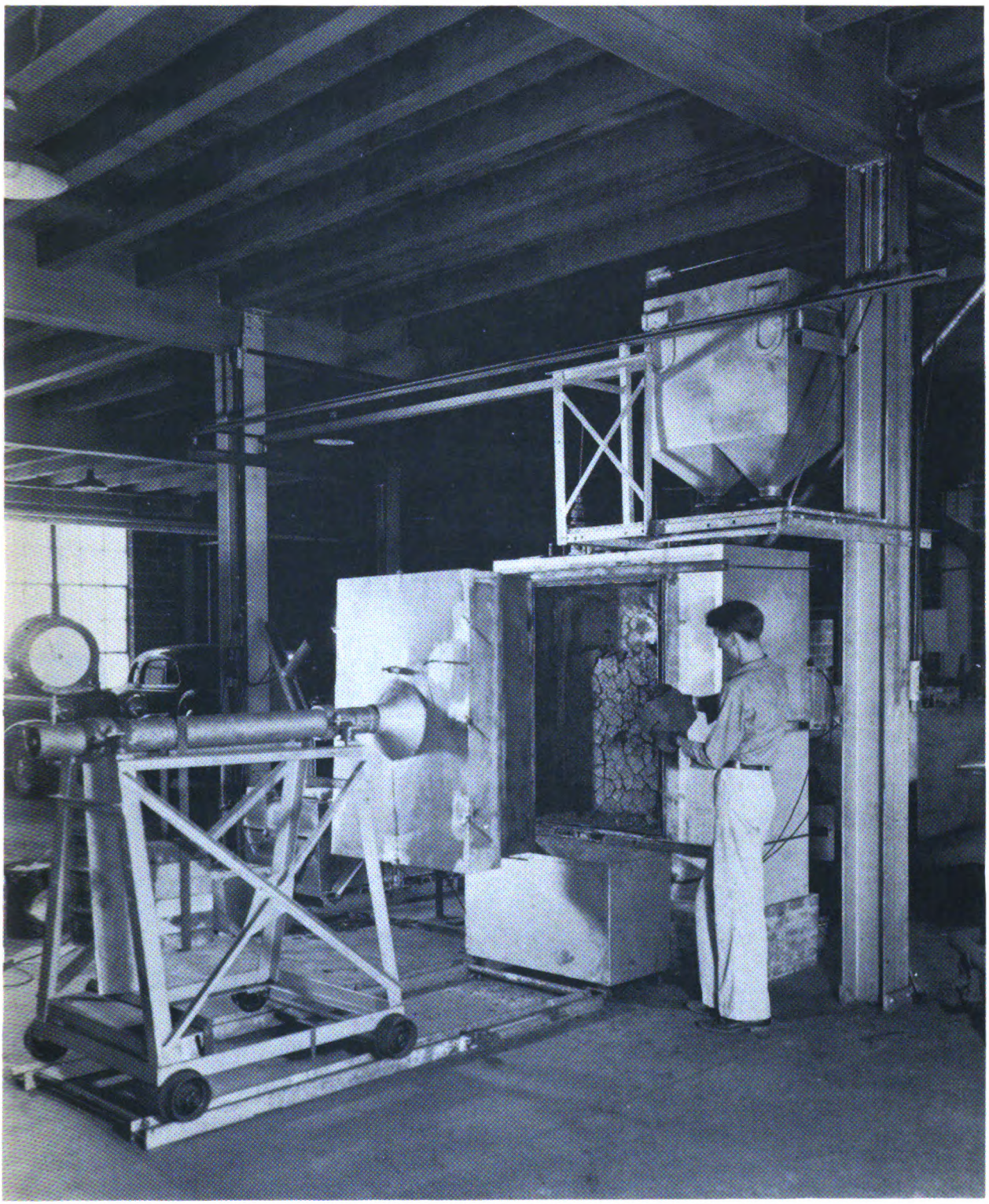

Figure 18.– Slot–type expansion oven (open).

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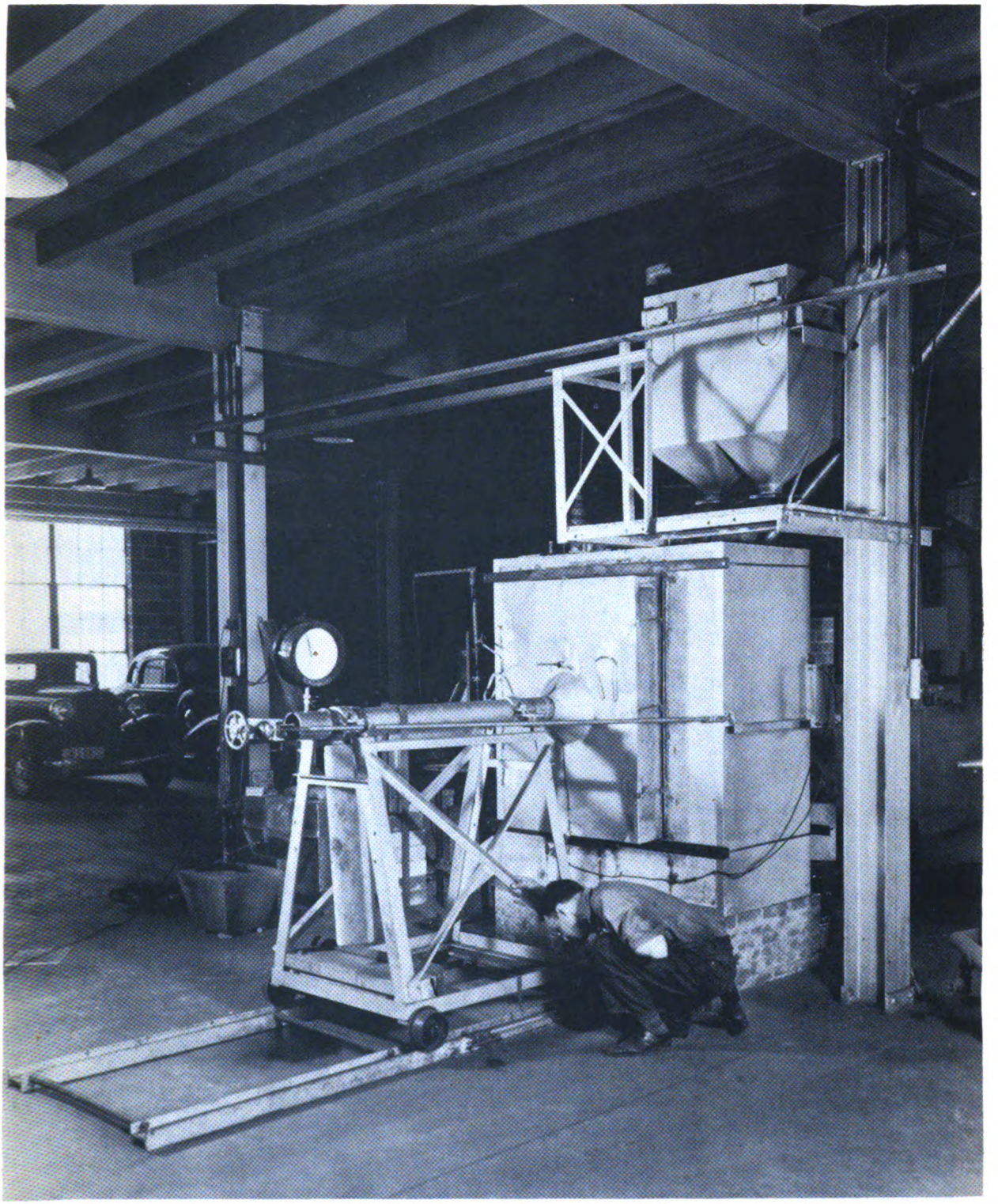

Figure 19.- Slot-type expansion oven (closed).

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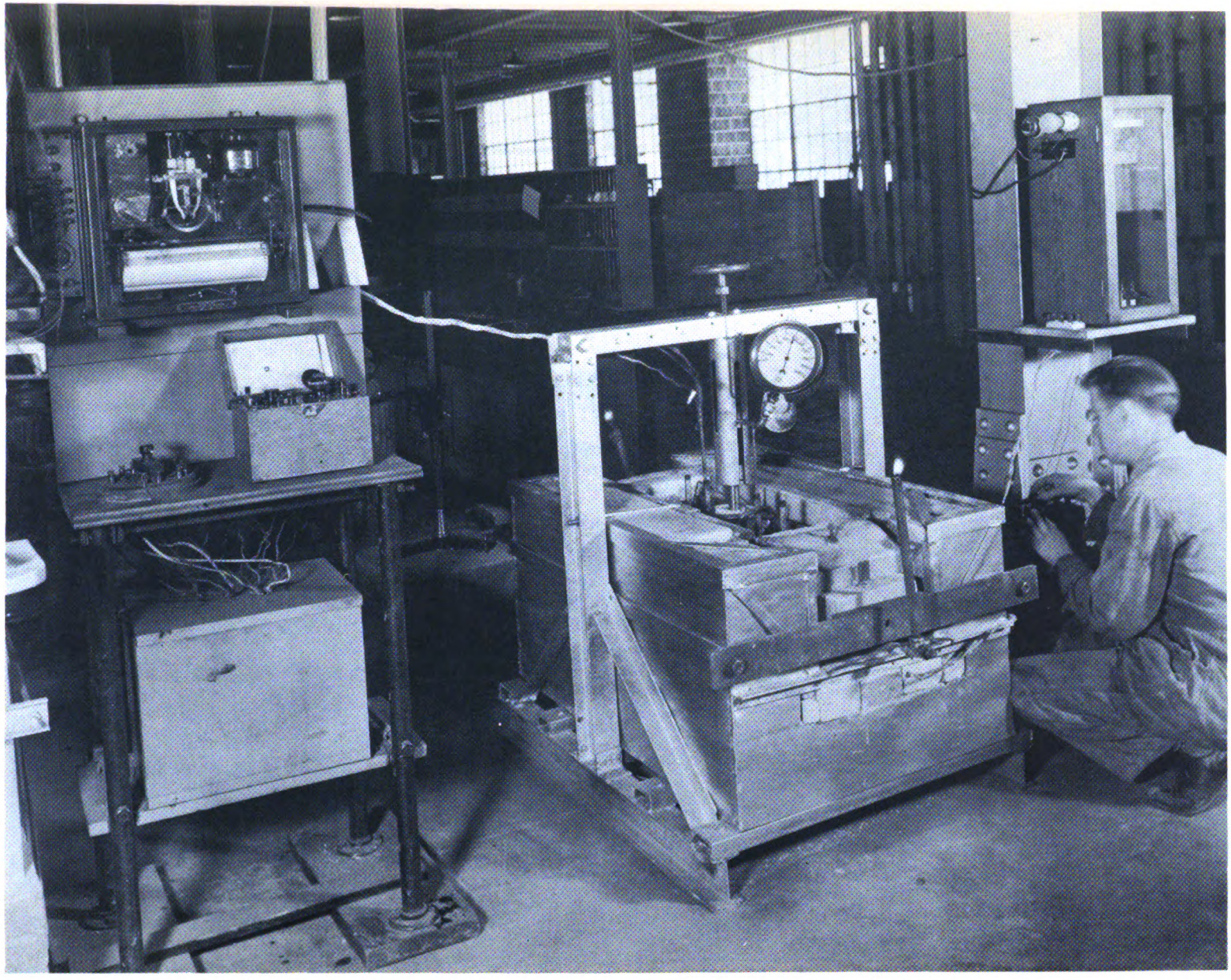

Figure 20.- Sole-heated expansion oven.

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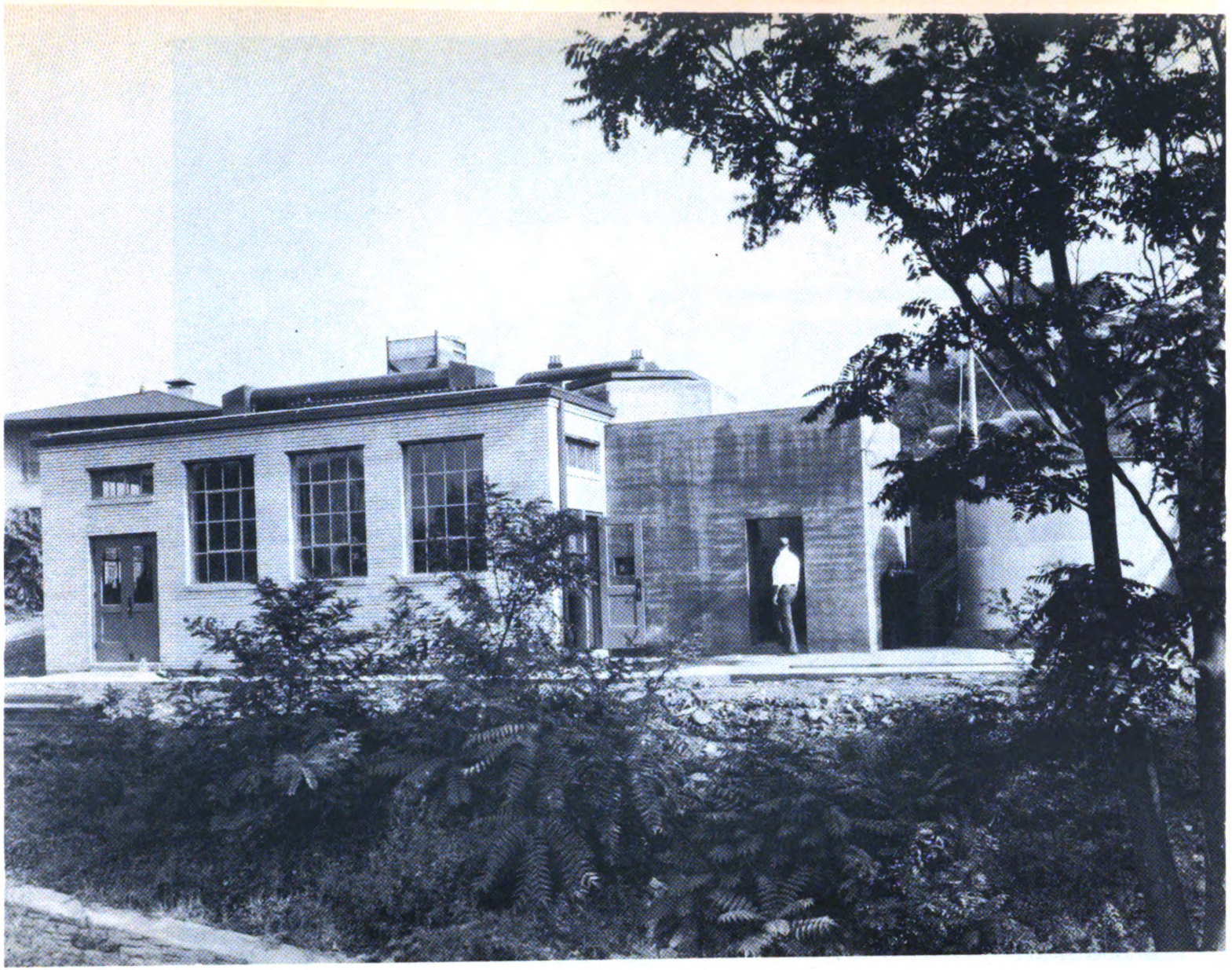

Figure 21.- Coal hydrogenation building.

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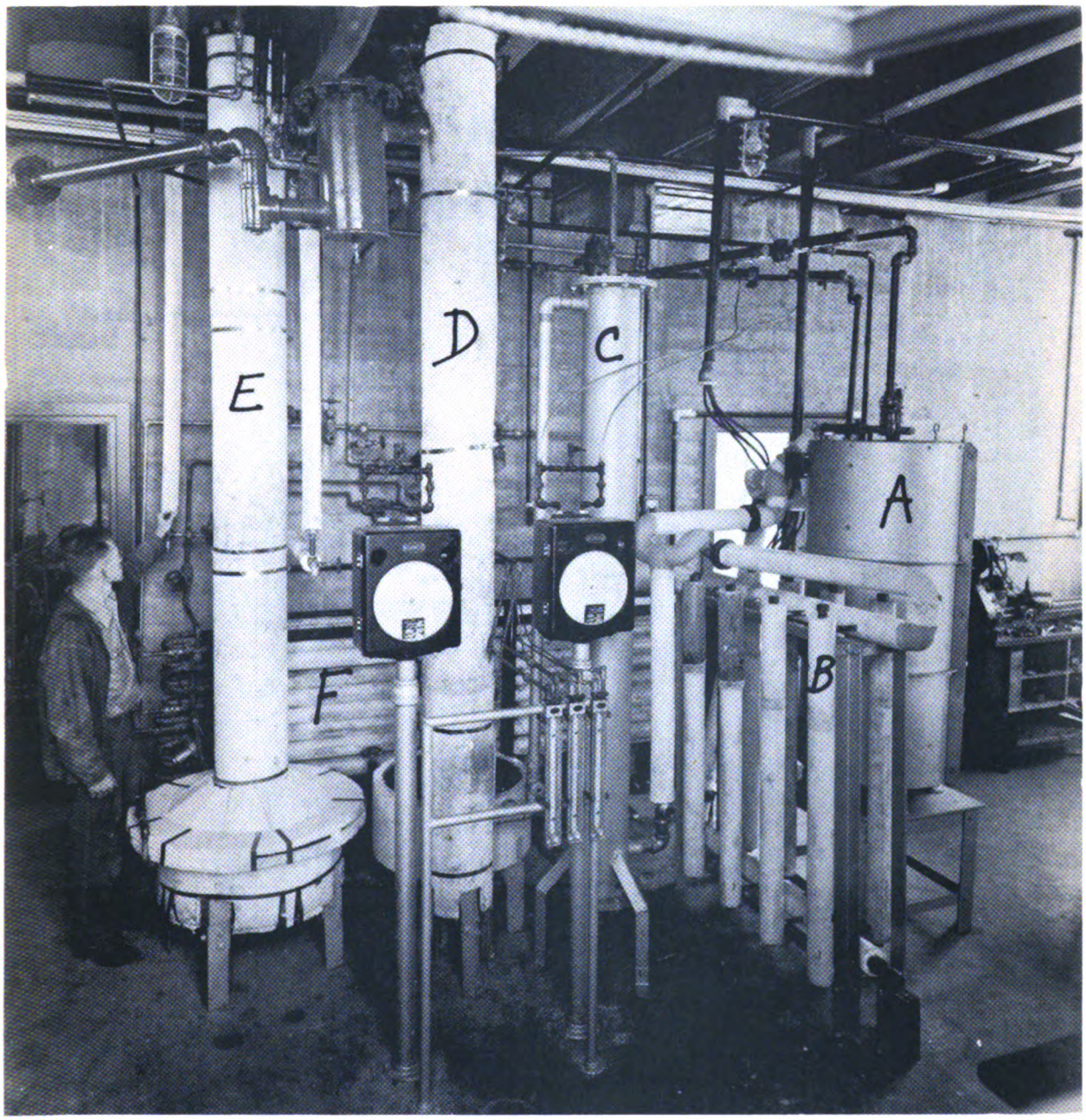

Figure 22.- Hydrogen plant.

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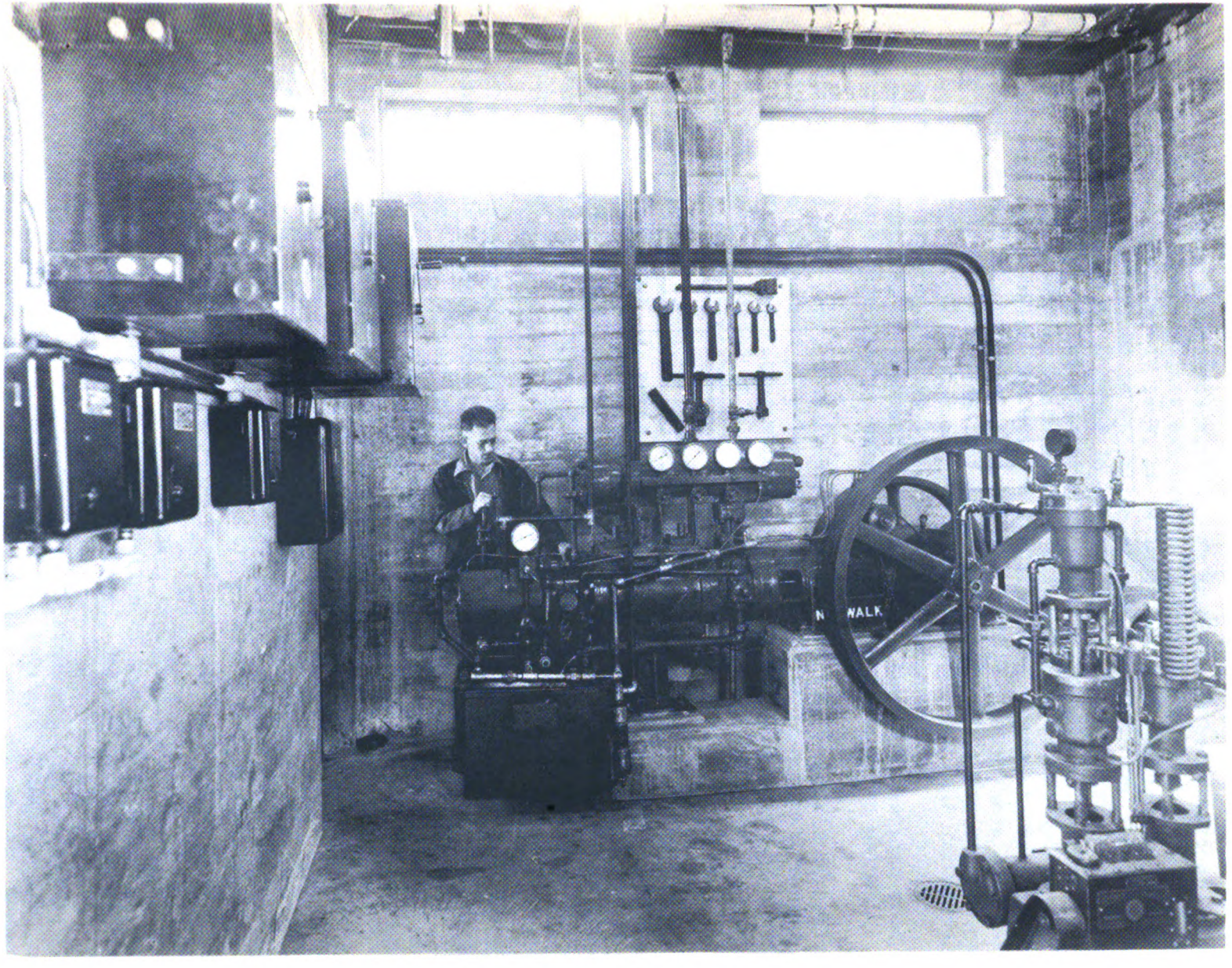

Figure 23.– Hydrogen compressor.

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Hydrogenation of Coal

In European and Asiatic countries much emphasis is being laid on the importance of providing against exhaustion of liquid fuel supplies. Although necessity in this country is less, it is important that American research in this field be maintained to establish proper groundwork for the evaluation of foreign developments. Another important objective of the study on the hydrogenation of coal is the obtaining of comparative data on various American coals to determine which are the most amenable to the hydrogenation treatment and to determine the optimum conditions of treatment for each. Heretofore, almost all hydrogenation studies in this country have been made by the oil industry. Investigations to be conducted by the Bureau of Mines will be dedicated to public use and for the benefit of the coal industry.[34/] By starting the program at this time, it is contemplated that during the next decade enough new developments will result so that the American coal industry will be in position to make liquid fuel when necessity arises.

During the past year, a small-scale continuous plant capable of hydrogenating 100 to 200 pounds of pulverized coal in 24 hours was put in operation. This equipment, together with apparatus for the production of hydrogen from natural gas, is housed in a special building at the Bureau's Central Experiment Station at Pittsburgh, Pa. (see fig. 21).

The hydrogen plant (fig. 22) consists of a water gas generator, A, water gas shift chamber, B, water spray tower, C, carbon dioxide scrubber, D, and amine regenerator, E. A 4-stage horizontal compresor (fig. 23) is used to compress the hydrogen. Hydrogen is stored in a battery of 25 high-pressure steel cylinders each of 200 cubic feet (measured at atmospheric pressure) capacity.

The water gas generator consists of two concentric tubes 7 inches and 5 inches outside diameter and 60 inches long, made of high chromium-nickel alloy (25 percent Cr, 20 percent Ni). The 1 inch annulus between these tubes is filled with pure nickel stampings 1/4 by 1/8 inch, which serve as catalyst for the reaction between the steam and natural gas. The reaction zone is maintained at 1,050° C. by two sources of heat, namely, a 1-1/2 inch Globar unit in the center of the inner tube and a high-temperature

34/ Fieldner, A. C., Oil from Coal: Science Digest, February 1937, pp. 68-70.
Chemical and Metallurgical Engineering, U. S. Looks into Coal Hydrogenation: vol. 44, no. 9, September 1937, p. 472.
Storch, H. H., Coal Hydrogenation. Chemistry: Ind. and Eng. Chem., Ind. Ed., vol. 29, December 1937, pp. 1367-1371.
Fisher, C. H., and Eisner, Abner, Primary Liquefaction of Coal by Hydrogenation. Destructive Hydrogenation of the Oxygen, Nitrogen, and Sulphur Linkages: Ind. and Eng. Chem., Ind. Ed., vol. 29, no. 12, December 1937, pp. 1371-1376.
Storch, H. H., Hirst, L. L., Golden, P. L., Pinkel, I. I., Boyer, R. L., Schaeffer, J. R., and Kallenberger, R. H., Bureau of Mines Experimental Plant: Ind. and Eng. Chem., Ind. Ed., vol. 29, no. 12, December 1937, pp. 1377-1380

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resistance heater surrounding the outer tube. Equal volumes (about 100 cubic feet per hour) of steam and natural gas are used. The analysis of the natural gas varies somewhat with time but averages about 90 percent CH_4, 8 percent C_2H_6, and 1.5 percent N_2. The water gas produced by the generator contains about 75 percent H_2, 21 percent CO, 1 percent CO_2, and 1 percent CH_4.

The water gas shift tubes, B, in figure 20 have been replaced by a more compact catalyst chamber to reduce heat losses. The new water gas shift chamber consists of a 24-inch section of 6-inch steel pipe containing at its center a 2-inch copper rod. Attached to the latter are eight copper plates or vanes, which divide the chamber into eight catalyst spaces. This arrangement serves to permit rapid conduction of heat and avoids excessive channeling of the mixture of water gas and steam. A copper-cobalt catalyst, the preparation of which has been described in a recent publication[35], is used to accelerate the water gas reaction: $H_2O + CO = CO_2 + H_2$, the temperature being maintained at about 350° C. Approximately five parts of steam are mixed with the water gas as it issues from the water-gas generator. The exact amount of steam added is determined by the desired temperature gradient in the column of copper-cobalt catalyst. The gradient is preferably kept below 50° C., that is, catalyst temperature 300° to 350° C.

The water-spray tower, C, serves to condense the excess steam in the off-gas from the cobalt-copper catalyst. The carbon dioxide scrubber, D, is a 10-inch by 12-foot tower packed with 1/4-inch carbon Rashig rings. A spray of 10 percent tetramine is used to scrub out the carbon dioxide.[36] The amine regenerator, E, consists of a boiler, 12 by 30 inches, attached to a distillation column 10 inches by 11 feet, packed with 1/4-inch carbon Rashig rings. The spent amine is heated in the heat exchanger, F, by transfer of heat from regenerated amine and is then pumped through a spray head in the center of the distillation column of E. A reflux coil carrying cooling water is in the top of this column.

The hydrogen plant has functioned satisfactorily during the production of about 6,000 cubic feet of hydrogen. The analysis of the hydrogen produced is approximately as follows: 96.0 percent H_2, 0.5 percent CO, 0.2 percent CO_2, 1.2 percent CH_4, 0.2 percent O_2, 1.9 percent N_2.

The coal hydrogenation plant is shown in figures 24, 25, 26. The coal-oil paste is delivered under several pounds pressure by the gear-type oil pump A to the paste pump, B (fig. 24). The latter is a vertical piston pump with ball check valves. The paste is forced into the converter, shown in figure 25, and contained in the furnace of figure 26, a 5-inch outside diameter by 3-inch inside diameter, 18 percent Cr, 8 percent Ni, alloy-steel

35/ Storch, H. H., and Pinkel, I. I., Preparation of an Active Cobalt-Copper Catalyst for the Water-Gas Shift Reaction: Ind. and Eng. Chem., Ind. Ed., vol. 29, 1937, p. 715.

36/ Hirst, L. L., and Pinkel, I. I., Absorption of Carbon Dioxide by Amines: Ind. and Eng. Chem., Ind. Ed., vol. 28, no. 11, November 1936, pp. 1313-1315.

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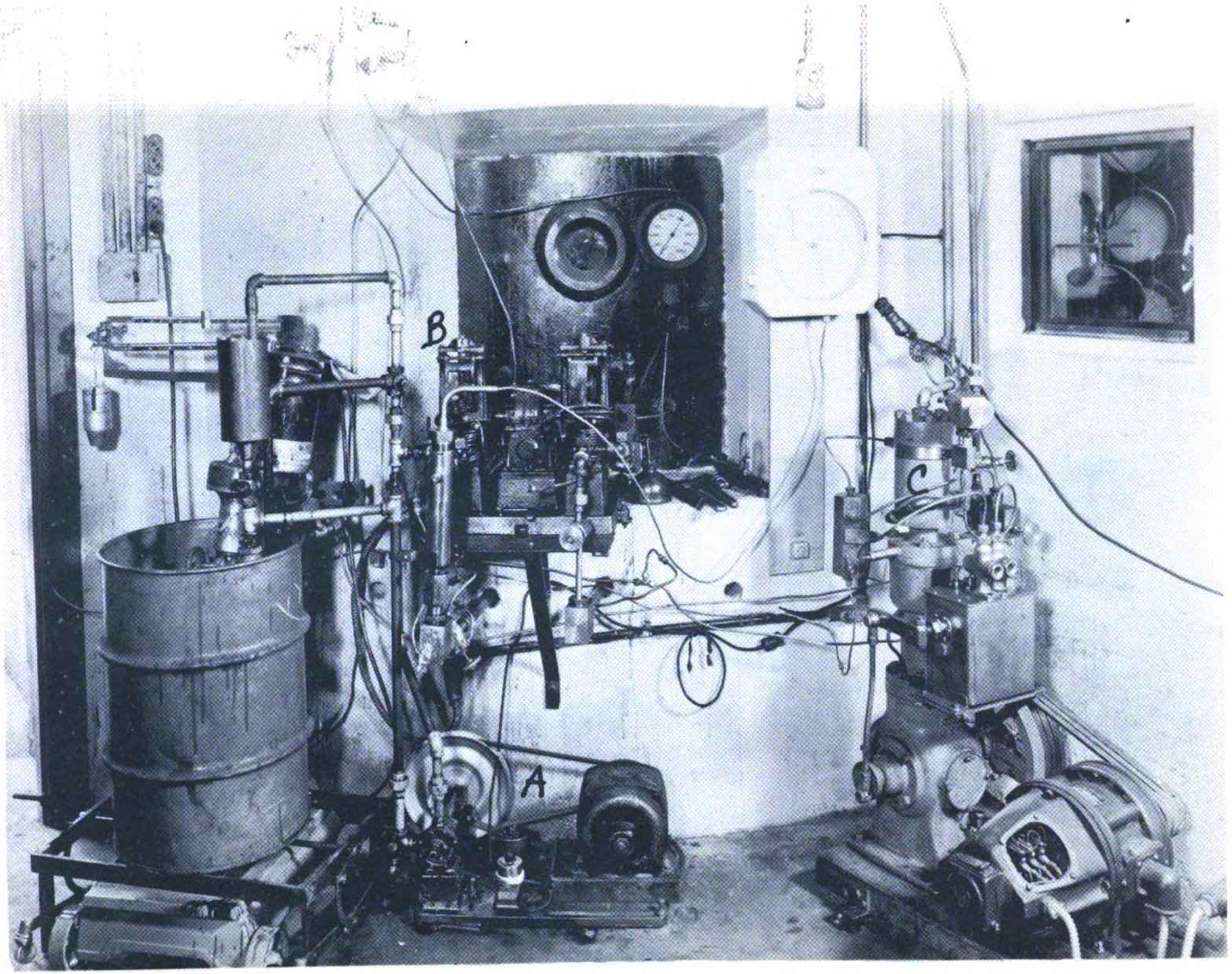

Figure 24.- Paste pumping and hydrogen recirculation.

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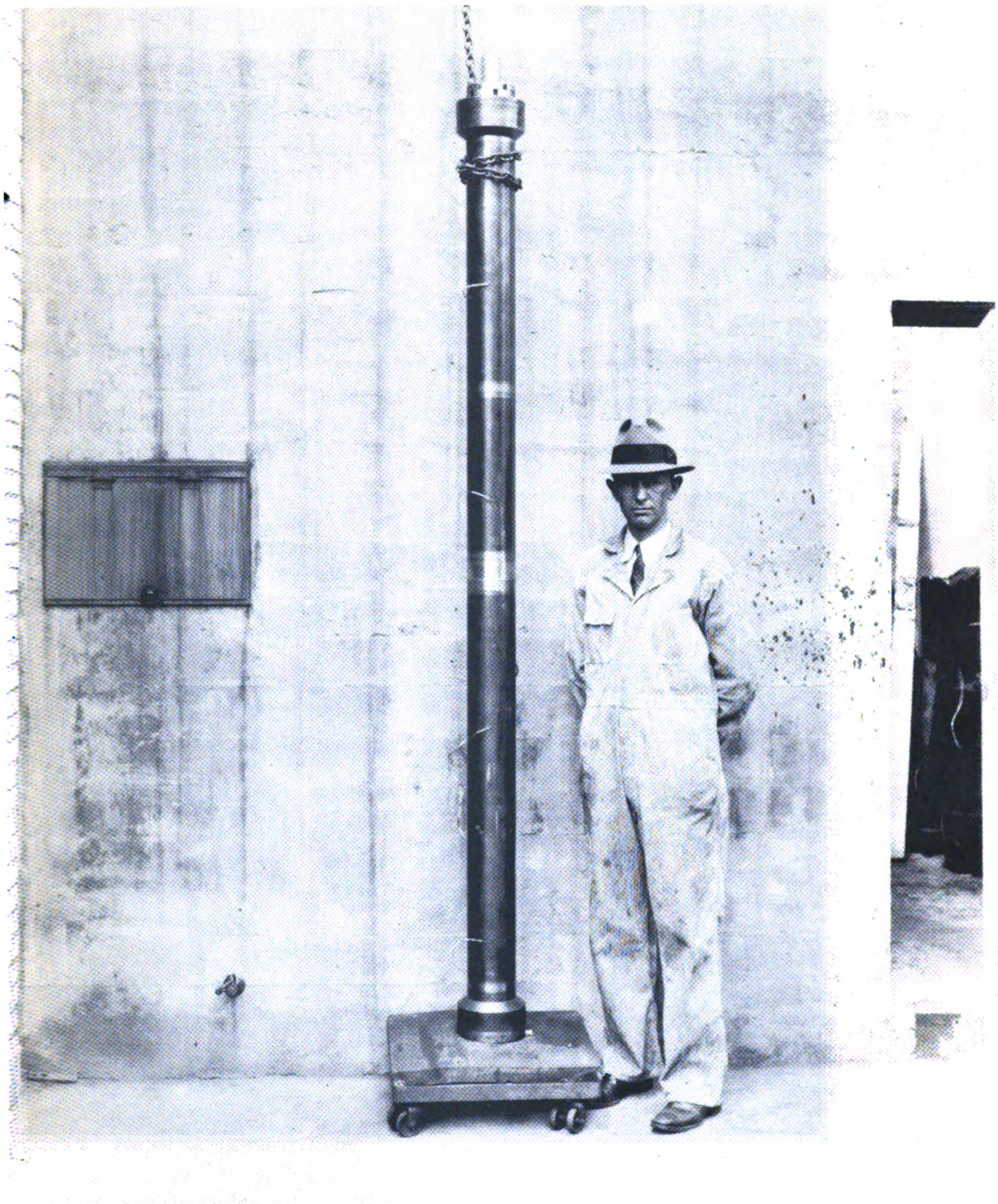

Figure 25.- Coal hydrogenation converter.

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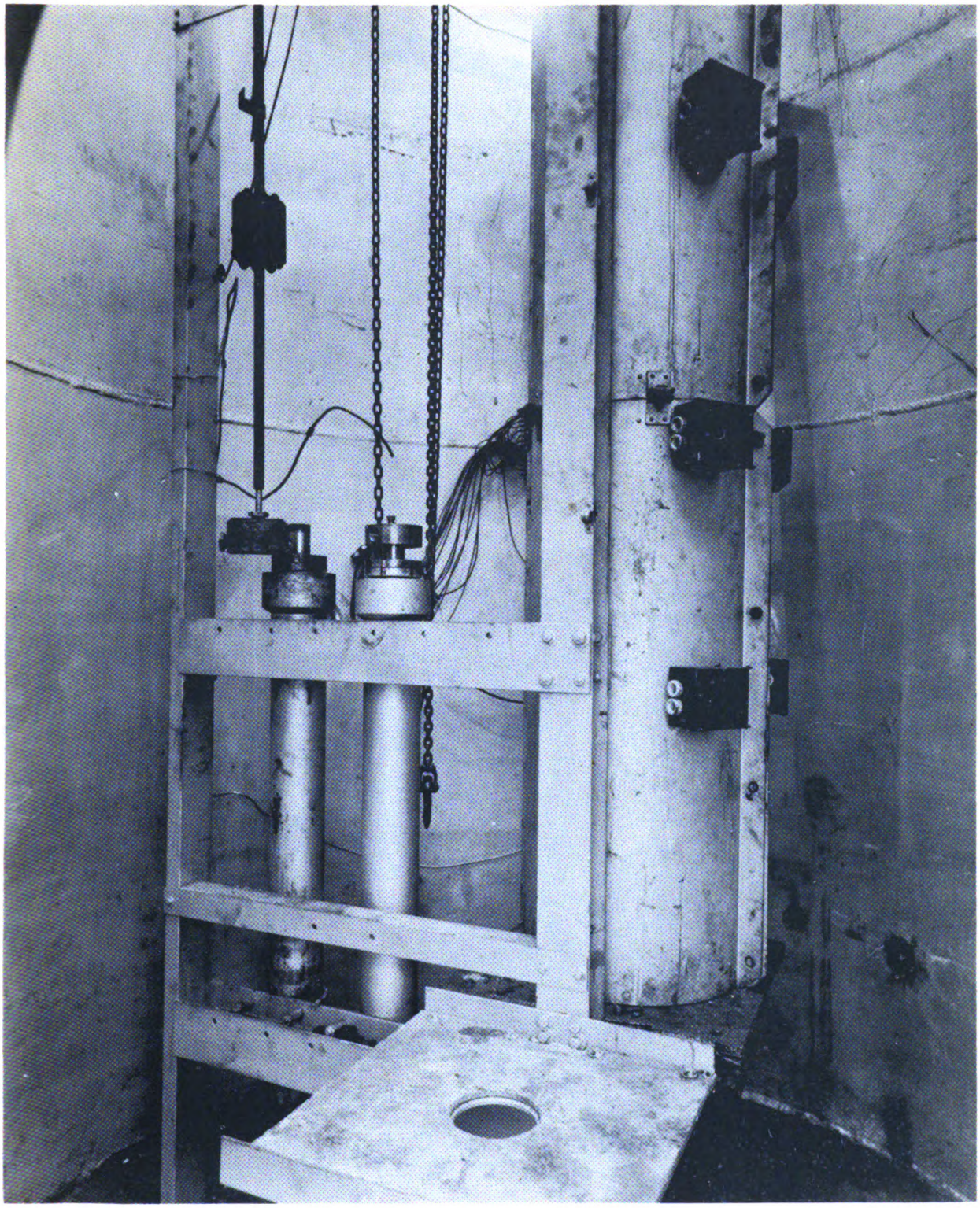

Figure 26.- Condensation traps and furnace.

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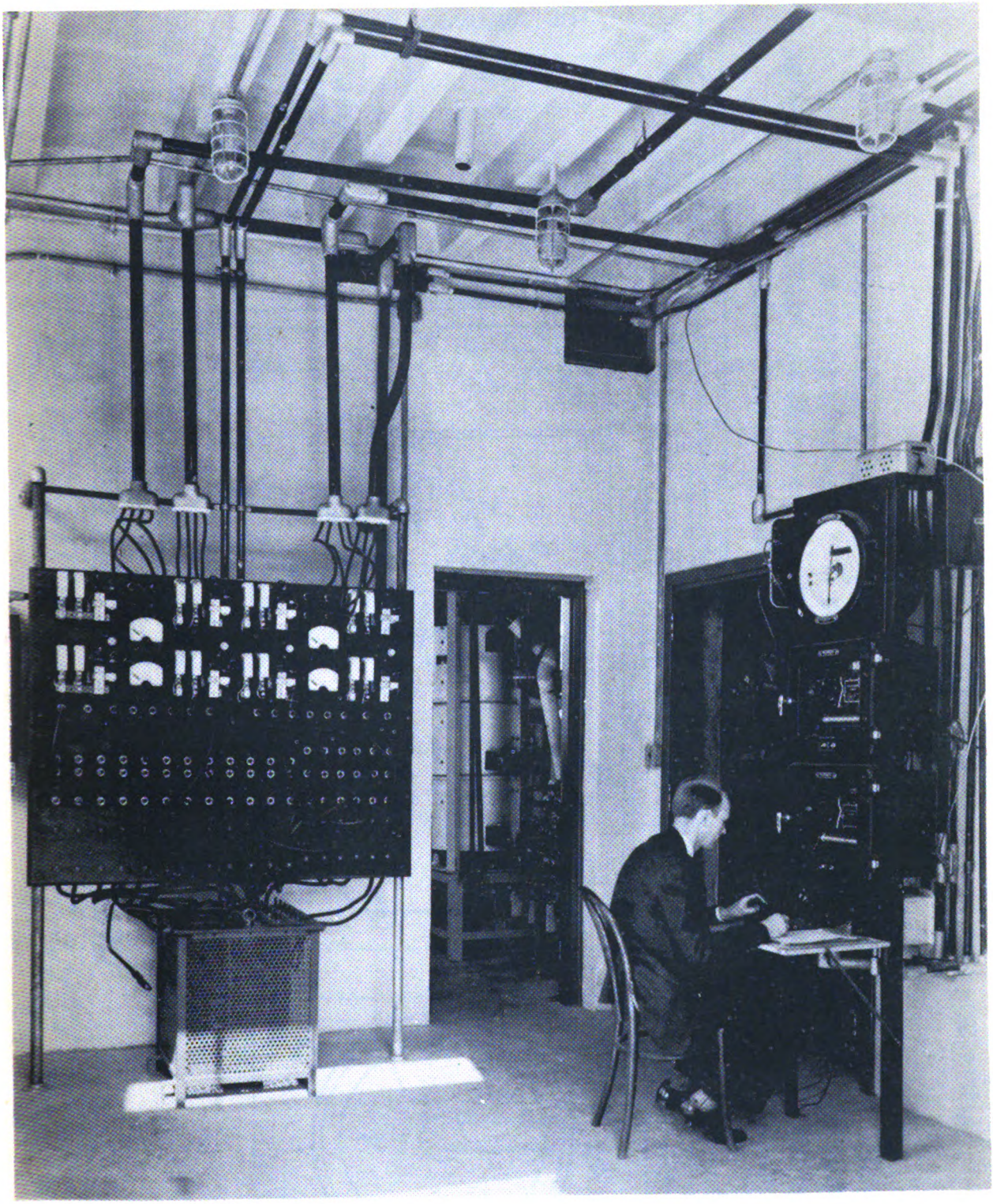

Figure 27.- Switchboard and recording instruments.

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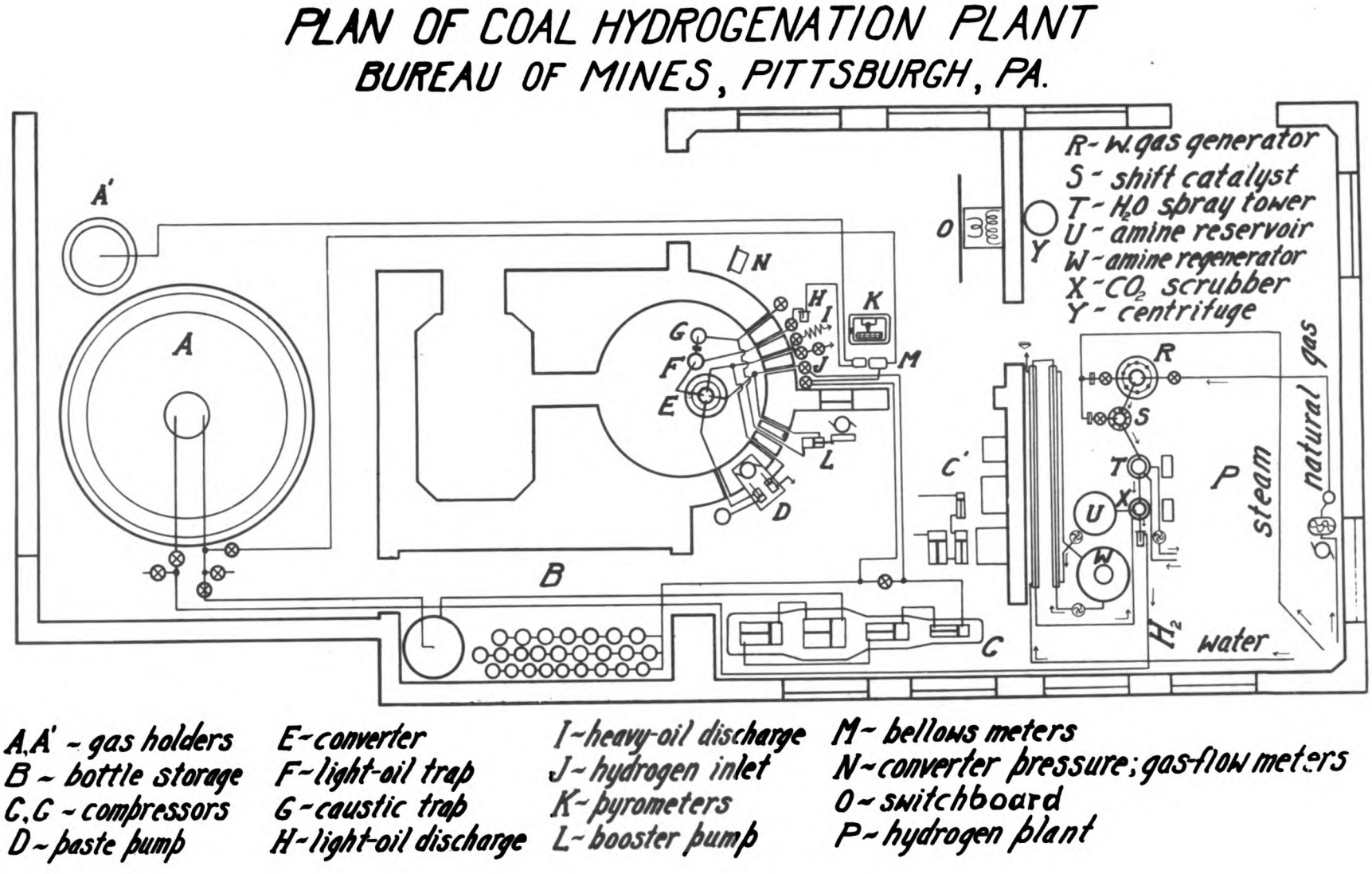

Figure 28.- Plan of coal hydrogenation plant, Bureau of Mines, Pittsburgh, Pa.

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tube. Hydrogen is pumped in through the top of the converter and preheated by passage through a heat exchanger. The latter consists of several coils of aluminum tubing through which the incoming hydrogen passes before being mixed with the paste. The light oil and hydrogen off-gas pass around these coils before leaving the converter. Heat is supplied to the converter by a cylindrical electric resistance furnace shown in figure 26. Temperatures are measured by thermocouples inserted in a well attached to the top closure of the converter and extending the full length of the latter. Surface temperatures on the outer wall of the converter are measured at three equidistant points.

The light oil and hydrogen off-gas pass through the two traps shown in figure 26. The light oil condenses in the first trap, and hydrogen sulphide, water vapor, and carbon dioxide are removed from the hydrogen gas by a layer of caustic soda in the second trap. From the latter the gas is picked up by the recirculation pump, C, (fig. 24), and pumped back into the converter.

A view of the switchboard and temperature-recording instruments is given in figure 27, and a floor plan of the entire plant in figure 28.

Six single-stage runs of 10 to 18 hours' duration have been made with Pittsburgh bed coal from the Bureau's Experimental Mine at Bruceton, Pa. These tests show that it is possible to liquefy 75 percent (ash- and moisture-free basis) of the coal, 22 percent being converted to normally gaseous hydrocarbons. The product consists of 50 percent of "light" oil, distilling mainly between 180° and 300° C., and 50 percent of a heavy oil, which resembles a low-temperature tar. This heavy oil is centrifuged to remove the ash and other solid matter, such as fusain and catalyst material, and the liquid is then used to make paste with an additional quantity of coal. The light oil is to be sent to the second hydrogenation stage, where it will be converted to gasoline. As an example of the data collected, tables 10 and 11 give the details of run no. C-4.

Effect of Vehicle in Coal Hydrogenation

The effect of vehicle in coal liquefaction, previously given comparatively little attention, was studied under mild conditions of hydrogenation (400° C. and 67 atmospheres original hydrogen pressure). Batch experiments were made with a small autoclave. Twenty-one vehicles, consisting of a single organic compound or a mixture of two were investigated. The same coal, Pittsburgh bed, obtained from the Bruceton mine, was used throughout.

The organic compounds employed were selected on the basis of certain outstanding properties, such as polarity, aromaticity, paraffinicity, depolymerizing action on coal, and tendency to lose hydrogen at high temperatures. With the results obtained, it is possible to draw several conclusions regarding the requirements of a satisfactory vehicle.

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TABLE 10. - Operating data from run no. C-4

Temperature: 410° C.; pressure: 3,200 lbs/sq. in.; H_2 recirculation rate: 200 to 300 cu. ft. (atmospheric pressure) per hour; total time of run: 18 hours; time of contact: 3 hours.

	Total weight, pounds	Total percent of ash- and moisture-free coal
Paste	94.0	----
Coal	46.5	----
Hydrogen absorbed	2.7	6.3
Liquid products	88.3	----
Gaseous products	8.4	19.6
Heavy oil	50.1	----
Light oil	34.5	----
Water	2.7	4.7
Solids	3.5	----
Oil yield	----	74.7

TABLE 11. - Analytical data on products

	Light oil		Vehicle (anthracene oil)		Heavy oil	
	Percent	Specific gravity	Percent	Specific gravity	Percent	Specific gravity
Sample as received	--	1.013	--	1.103	--	--
Distilling under 200° C.	18.5	.880	1.5	--	3.0	--
Distilling, 200°-270° C.	45.0	.993	30.5	Solid	--	--
Distilling, 270°-300° C.	16.5	1.054	15.0	1.090	--	--
Distilling, 300°-330° C.	10.0	1.075	14.0	Solid	--	--
Distilling, over 330° C.	10.0	--	39.0	--	--	--
Distilling to 200°C. at 5 mm	--	--	--	--	53.0	1.057
Tar bases	4.2	--	6.4	--	3.8	--
Phenols	9.2	--	4.4	--	7.4	--
Caboxylic acids	.4	--	.0	--	.2	--
Neutral oil	86.2	.990	89.2	1.064	--	--
Olefins, percent of neutral oil	7.5	--	8.0	--	--	--
Aromatics, percent of neutral oil	82.6	--	92.0	--	--	--
Saturates, percent of neutral oil	9.9	--	.0	--	--	--
Acetone insoluble	--	--	--	--	6.7	--
Tetralin insoluble	--	--	--	--	5.7	--

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Previous claims that tetralin is a good dispersion medium were confirmed. The only other hydrocarbon (methylnaphthalene) observed to be effective is similar to tetralin in that it is partly aromatic and partly aliphatic. Completely aromatic hydrocarbons (naphthalene and diphenyl) and saturated hydrocarbons (decahydronaphthalene and hexadecane) proved to be poor vehicles. The presence of polar compounds, such as phenols and amines, in the vehicles was found to be beneficial.

The products formed in highest yield were pitches, which melt at moderately elevated temperatures. Elementary analyses of these pitches indicated that hydrogenation had raised the carbon and hydrogen contents, lowered the sulphur and oxygen contents, and caused little change in the nitrogen content. The results of this work have been published.[37]

Batch experiments are in progress on the rate of destructive hydrogenation of functional groups containing oxygen, nitrogen, and sulphur in the coal substance. It is expected that some fundamental information concerning the chemistry of the coal-hydrogenation reactions will result from this work.

Analytical Methods for Coal Hydrogenation Products

A survey was made of the sources of error involved in extraction procedures for the analysis of tar oils.[38] After a series of experiments, a modification of the present Bureau of Mines method for analysis of tar oils was developed. The important changes are:

1. A correction for the water absorption by the tar oil in the first extraction.

2. The separate determination of phenols and carboxylic acids.

The modified method is simpler and more accurate than the methods previously used to determine tar acids, tar bases, and carboxylic acids.

A similar series of experiments was made in order to determine the limits of error in sulphuric acid-extraction methods for determining olefins and aromatics in hydrocarbon oils. None of these methods gives results applicable with accuracy to all mixtures of hydrocarbons. It was found, however, that for the neutral oils found in coal tar and related oils the Bureau of Mines' method developed by Kester and Pohle[39] gave the most

37/ Fisher, C. H., and Eisner, Abner, Primary Liquefaction of Coal by Hydrogenation. (Chemical Nature and Effectiveness of the Vehicle): Ind. and Eng. Chem., Ind. Ed., vol. 29, no. 8, 1937, pp. 939-945.

38/ Fisher, C. H., and Eisner, A., Extraction Methods for Determining Tar Acids and Bases, and Variables Affecting Their Accuracy: Rept. of Investigations 3310, Bureau of Mines, 1937, 34 pp.
Fisher, C. H., and Eisner, A., Determination of Tar Acids and Bases by Extraction Methods: Ind. and Eng. Chem., Anal. Ed., vol. 9, no. 5, 1937, pp. 213-218.

39/ Kester, E. B., and Pohle, W. D., Determination of Olefin, Aromatic and Paraffin Hydrocarbons in Neutral Oil from Coal Tar: Ind. and Eng. Chem., Anal. Ed., vol. 3, 1931, pp. 294-297.

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accurate results. A description of the experimental work involved in this survey will be published at an early date[40].

In the usual procedures used for the analysis of neutral oils, the olefin content is estimated from the volume of residual oil after extraction with 80 to 90 percent sulphuric acid and the aromatic content from the volume after extraction with 96 to 100 percent acid. Although these procedures have been studied extensively and various improvements developed, it is generally agreed that methods of this kind are not entirely satisfactory.

Subsequent to the survey described above, relating to sulphuric extraction procedures for neutral oil analysis, Fisher and Eisner[41] developed a new method that is much more accurate than those used heretofore. This new method is based on the changes in physical constants (refractive index and specific gravity) of the oil which accompany the stepwise removal of olefins and aromatics as effected by successive extractions with several sulphuric acid solutions of increasing concentration. The curve obtained by plotting the specific refraction against the volume of residual oil shows a maximum when the olefins have been completely removed and a minimum when all of the aromatics have been extracted[42]. Various hydrogenated coal distillates, two synthetic mixtures (whose analysis by the older methods always yielded inaccurate results), and two gasolines were analyzed more accurately than has been possible heretofore.

Pyrolysis of Ethane

A study of the rate and mechanism of thermal decomposition of ethane was made at pressures from 10 to 140 cms. of Hg. and at two temperatures, namely, 541° and 565° C. These experiments were static; the ethane being confined in a 500-cc bulb made of a special glass, which did not deform under the experimental conditions. Pressure measurements were made every 2 minutes and chemical analyses of the reaction mixture were done at the end of 30, 60, and 120 minutes.

The production of methane during the pyrolysis of ethane was traced to two reactions:

$$2C_2H_6 = 2CH_4 + C_2H_4$$

and $$C_2H_6 + C_2H_4 = CH_4 + C_3H_6.$$

40/ Fisher, C. H., and Eisner, A., Sulphuric Acid Extraction Methods for Determining Olefins and Aromatics in Hydrocarbon Oils. Optimum Conditions and Concentrations of Acid: In press, to be published as Report of Investigations, Bureau of Mines.

41/ Fisher, C. H. and Eisner, A., Analysis of Neutral Oils by Sulphuric Acid Extraction. Use of Physical Constants for Analytical Purposes: To be published in Ind. and Eng. Chem., Anal. Ed.

42/ Fisher, C. H., and Eisner, A., Graphic Analysis of Hydrocarbon Oils: Ind. and Eng. Chem., Anal. Ed., vol. 29, no. 31, Aug. 15, 1937, pp. 366-370.

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The rates of these reactions were measured. There is no measurable production of methane by the reaction $H_2 + C_2H_6 = 2CH_4$. The reaction between ethane and ethylene to produce methane and propylene will be of importance in polymer gasoline production from natural gas and refinery gases, for the polymerization of propylene is a much simpler task than that of ethylene.

The initial rate of dehydrogenation of ethane was found to be first order from 200 to 1,400 mm. of Hg. pressure. The rate during an experiment is well represented by the equation:

$$dH_2/dt = k(C_2H_6) - k'(H_2)\ (C_2H_4)$$

The applicability of a free radical chain mechanism to these experimental results has been investigated. There are two serious objections to such a mechanism for the entire reaction, but it is possible for chain reactions to contribute a small percentage to the total.

Our own experiments and recalculation of Pease and Durgan's[43] data in the light of our work on methane production suggest a lower value for the equilibrium constant of the dehydrogenation reaction than has been obtained in previous experimental work. This new value is in far better agreement with statistical mechanical calculations than were the earlier values.

The results of the ethane work and other theoretical investigations have been published.[44]

Activated Adsorption of Hydrogen By Coal Hydrogenation Catalysts

Two theoretical analyses of existing data on the activated adsorption of hydrogen have been made.[45]

43/ Pease, R. N., and Durgan, E. S., Equilibrium in the Reaction $C_2H_6 \rightleftharpoons C_2H_4 + H_2$ Jour. Amer. Chem. Soc., vol. 50, 1928, pp. 2715-2718.

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The analysis of Burwell and Taylor's[46] data on the activated sorption of hydrogen by chromium oxide led to the derivation of a rate equation that fits the data for moderate amounts of sorption very well. The derivation is based on a simple model of the sorption process involving primary adsorption followed by diffusion to secondary centers. This model also accounts, qualitatively at least, for the failure of the equation at extreme conditions.

In the second paper,[47] it was shown that the assumption of dissociative adsorption on a uniform (Langmiur-type) surface leads to equations that account for the observed equilibria in the primary activated adsorption of hydrogen on copper. A discussion is presented of the possibility of extension of these equations to rates of adsorption on copper and on other metals.

An apparatus has been constructed for studying the rates of activated adsorption of hydrogen as a function of temperature and pressure, and it is being standardized with a copper catalyst before proceeding with coal-hydrogenation catalysts. This work will be of importance in the selection of vapor-phase catalysts for the hydrogenation of the middle oils from the first stage of coal hydrogenation.

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47/ See footnote 45, second reference.

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